

MORCZCV, I. S.

PA 63/49T9

USSR/Chemistry - Manganese Oxides Jul/Aug 49

"Gamma-Modification of Manganese Dioxide," I. S. Morozov, V. G. Kuznetsov, Inst of Gen and Inorg Chem iment N. S. Kurnakov, Acad Sci USSR, 11 pp

"Iz Ak Nauk SSSR, Otdel Khim Nauk" No 4

Chlorination of beta-Mn₂O₃ in a water medium with chlorine gas produced the subject modification which was then investigated by X-ray (photographs included) and thermal analysis, and also as to its specific weight. Results showed it to be a polymorphic modification of beta-MnO₂, and that conversion of gamma-MnO₂ into beta-MnO₂

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USSR/Chemistry - Manganese Oxides Jul/Aug 49
(Contd)

proceeded irreversibly at 325°, accompanied by a decrease in specific volume and a low-heat liberation. Adsorption of cations by gamma-MnO₂ and beta-MnO₂ powders from electrolyte solutions is the same for equal dispersion of particles. Modification can be used as a depolarizer in galvanic cells. Submitted 17 Jul 48.

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MOROZOV, I. S. and KORSHUNOV, B. G.

"An investigation of the chlorination reaction of niobium pentoxide zirconium dioxide", Khimiya Redkikh Elementov, No. 2, p 102, 1955.

The above reaction for Nb O was investigated in the temp. region 700-1100°C. Vapour pressure and the stability niobium chlorooxide were determined. The equilibrium constant of the reaction: $2\text{Nb}_2\text{O}_5 + 4\text{NbOCl}_3 \rightleftharpoons 6\text{Cl}_2 + 3\text{O}_2$ and standard free energy changes at temp. 800, 900, 1000 and 1100°C. Chlorination of ZrO₂ was investigated in the temperature region 1000-1200°C. The equilibrium constant of the reaction $\text{ZrO}_2 + 2\text{Cl}_2 \rightleftharpoons \text{ZrCl}_4 + \text{O}_2$ as well as standard free energy changes at temp. 1000, 1100 and 1200°C were determined.

SO: D-413171

Inst. Sci. + Indus. Chem. in ...

MOROZOV, I. S.

USSR/Physical Chemistry - Thermodynamics. Thermochemistry. B-8
Equilibrium. Physicochemical Analysis. Phase Transitions

Abs Jour : Referat Zhur - Khimiya, No 2, 1957, 3744

Author : Morozov I.S., Korshunov B.G.

Title : Thermal and Tensimetric Study of System NbCl_5 - ZrCl_4 -
NaCl.

Orig Pub : Zh. neorgan. khimii, 1956, 1, No 1, 145-157

Abstract : By the method of thermal analysis a study was made of the interaction of NbCl_5 (I), NaCl (II) and ZrCl_4 (III) on their conjoint crystallization from melts. Fusion diagrams have been plotted for the binary systems: I-II, II-III and I-III. In the system I-II has been found the incongruently melting compound $\text{NaCl} \cdot \text{NbCl}_5$ (IV) with temperatures of peritectic and polymorphous transformations of 430° and 256° , respectively. II and III form a congruently melting (M.P. 695°) compound $2\text{NaCl} \cdot \text{ZrCl}_4$ (V) which exists in three modifications: $\overset{341}{\rightleftharpoons} \overset{377}{\rightleftharpoons} \overset{377}{\rightleftharpoons} \overset{377}{\rightleftharpoons}$.

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Moscow Inst. FINE Chem Technology in M.V. Lomonosov

/Morozov I.

USSR/Thermodynamics - Thermochemistry. Equilibria.
Physical-Chemical Analysis. Phase Transitions.

B-8

Abs Jour : Referat Zhur - Khimiya, No 6, 1957, 18509

Author : I.S. Morozov.

Title : Chemical Interaction of Niobium Hydroxide with Hydroxides
of Rare-Earth Metals, Titanium and Iron.

Orig Pub : Zh. neorgan. khimii, 1956, 1, No 4, 791-798

Abstract : The investigation of the character of the interaction
between the simultaneously precipitated hydroxides of
Nb and rare-earth metals, or Ti, or Fe^{2+} was carried
out by the thermal and roentgenphase analyses, as well
as by chemical methods - by chlorinating the oxydes at
650° with $TiCl_4$ (I) and their solution in hydrochloric
acid at heating. The thermographs of simultaneously
precipitated hydroxides Nb_2O_5 (II) and Nd_2O_3 taken in
the relation 3:1 and 1:1 show an exothermic effect (EE)
at 740° corresponding to the formation of a new phase,

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M. Morozov, I.S.

USSR/Physical Chemistry - Thermodynamics, Thermochemistry, B-8
Equilibria, Physical-Chemical Analysis, Phase Transitions.

Abs Jour : Referat Zhur - Khimiya, No 1, 1958, 405

Author : I.S. Morozov, B.G. Korshunov, A.T. Simonich.

Inst : Academy of Sciences of Ukrainian SSR, Chemical Institute.

Title : Thermal and Tensimetric Study of Systems $TaCl_5$ - $AlCl_3$ -
 $NaCl$ and $NbCl_5$ - $AlCl_3$ - $NaCl$.

Orig Pub : Zh. neorgan. khimii, 1956, 1, No 7, 1646-1657

Abstract : The binary systems $TaCl_5$ (I) - $NaCl$ (II), I - $AlCl_3$ (III)
and $NbCl_5$ (IV) - III were preliminarily studied thermally;
the systems II - IV (RZhKhim, 1957, 3744) and II - III
(I.I. Shvartsman, Zap. In-ta khimii AN UkrSSR, 1946, 7,
vyp. 1) were studied earlier. The formation of an inco-
gruently melting compound $NaTaCl_6$ (V) was revealed in the
system I - II, its temperature of peritectic transforma-
tion

Card 1/2

*INST Gen + Inorg Chem in N S KERNAROV
Moscow Inst. Fine Chem Technology*

USSR/Physical Chemistry - Thermodynamics, Thermochemistry, B-8
Equilibria, Physical-Chemical Analysis, Phase Transitions.

Abs Jour : Ref Zhur - Khimiya, No 1, 1958, 405

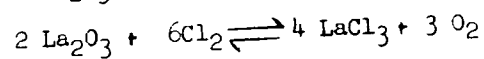
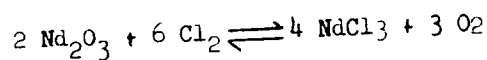
is 470° and its temperature of polymorphous transformation is 232°. The liquidus fields of the systems I - II - III and II - III - IV were plotted; the fields of primary crystallization of the compounds were determined. The tensimetric study of these systems by the flow method showed that the thermic stability of the chemical compounds V, NaNbCl_6 (VI) and NaAlCl_6 was not the same. The changes of free energy at the reactions of thermal dissociation of V and VI at 200 to 400° were determined using the obtained data. The formation of solid solutions I-II and III-IV was established tensimetrically.

Card 2/2

MOROZOV I. S.

"Interaction of the Oxides of Neodymium and Lanthanum With Gaseous Chlorine," by I. S. Morozov and B. G. Korshunov, Zhurnal Neorganicheskoy Khimii, Vol 1, No 11, Nov 56, pp 2606-2611.

The chlorination of the oxides of neodymium and lanthanum at the temperatures of 200°, 250°, and 300° was investigated. The equilibrium constants and changes of free energy in the reactions



were determined at these temperatures. In the introduction explaining the purpose of the work, it is pointed out that considerable interest in the chlorination processes and the properties of metal chlorides is evident in metallurgy at present, because chlorides can be prepared easily from metal oxides and other compounds, have low melting points, and are highly volatile, so that metals can be separated by taking advantage of this volatility. (U)

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MOROZOV, I.S.

Reaction of niobium and tantalum pentachloride salts with ferric
chloride and aluminum chloride. Zhur. neorg. khim. 1 no. 12:2792-
2802 D '56. (MLRA 10:6)

(Niobium chlorides) (Tantalum chloride)
(Systems (Chemistry))

MOROZOV, I.S.; TOPTYGINA, G.N.

Hydrochloric acid solutions of titanium chlorides. Zhur.neorg.
khim. 2 no.7:1629-1638 J1 '57. (MIRA 10:11)
(Titanium chlorides) (Hydrochloric acid)

MOROZOV, I.S.; SIMONICH, A.T.

Thermal and tensimetric analysis of the system $TaCl_5$ -- $AlCl_3$ --
CsCl. Zhur. neorg. khim. 2 no.8:1907-1914 Ag '57. (MIRA 11:3)
(Tantalum chlorides) (Aluminum chloride) (Cesium chloride)

MOROZOV, I.S.; TOPTYGIN, D.Ya.

Interaction between titanium tetrachloride and the chlorides of
tantalum, niobium, and aluminum. Zhur. neorg. khim. 2 no.8:1915-
1921 Ag '57. (MIRA 11:3)

(Chlorides) (Solubility)

MOROZOV, I.S.; TOPTYGIN, D.Ya.

Solubility of ferric chloride and its alloys with niobium
chloride, aluminum chloride, and sodium chloride in titanium
tetrachloride. Zhur.neorg.khim. 2 no.9:2129-2135 S '57.
(MIRA 10:12)

(Solubility) (Chlorides)

AUTHORS: Morozov, I.S., Toptygin, D.Ya.

TITLE: On the Interaction Between Ammonium Chloride and the Chlorides of Tantalum, Niobium, Titanium, Aluminum, and Iron
(O vzaimodeystvii khloristogo ammoniya s khloridami tantala, niobiya, titana, alyuminiya i zheleza)

PERIODICAL: Zhurnal neorganicheskoy khimii, 1958, Vol. 3, No. 7, pp. 1627-1631 (USSR)

ABSTRACT: The systems $TaCl_5-NH_4Cl$ and $NbCl_5-NH_4Cl$ were investigated by thermal analysis. The results of these investigations showed that in the system $TaCl_5-NH_4Cl$ the compound NH_4TaCl_6 exists with a congruent melting point at $304^\circ C$. NH_4TaCl_6 is of light yellow color. In the system $NbCl_5-NH_4Cl$ the compound NH_4NbCl_6 forms with an incongruent melting point at $205^\circ C$. This compound is of green color. The system $TiCl_4-NH_4$ assumes a bright yellow color when heated, and this is probably accompanied by the formation of ammonium hexachlorotitanate. Tensimetric analyses were carried out of the compounds NH_4AlCl_4 , NH_4TaCl_6 and NH_4NbCl_6 . The results obtained showed that each of the four double chloride compounds

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On the Interaction Between Ammonium Chloride and the
Chlorides of Tantalum, Niobium, Titanium,

478-3-7-30/4

behaves differently when heated. NH_4AlCl_4 sublimates without decomposition, but the compounds NH_4TaCl_6 and NH_4NbCl_6 are decomposed into their components when heated. There is no reaction between titanium tetrachloride and the compounds NH_4AlCl_4 , NH_4TaCl_6 and NH_4FeCl_4 . Heating of the above mentioned chlorides was found to be accompanied by the formation of layers of titanium tetrachloride in a wide interval of concentration. NH_4NbCl_6 decomposes completely when heated with titanium tetrachloride at a temperature of 250°C . It follows herefrom that a purification of titanium tetrachloride from chlorides of tantalum, aluminum and iron dissolved in it is possible with the aid of ammonium chloride. This method can, however, not be employed with success in the presence of NbCl_5 . There are 3 figures, 5 tables, and 10 references, 5 of which are Soviet.

SUBMITTED: May 31, 1957

1. Ammonium chlorides--Chemical reactions 2. 1957
--Chemical reactions

Card 2/2

AUTHORS: Sun Yin-chu, Morozov, I. S. SOV, 78-3-8-32, 48

TITLE: The Interaction Between the Chlorides of Rare Earth Metals and the Chlorides of Alkali and Alkaline Earth Metals
(Vzaimodeystviye khloridov redkozemel'nykh metallov s khloridami shchelochnykh i shchelochnozemel'nykh metallov)

PERIODICAL: Zhurnal neorganicheskoy khimii, 1958, Vol. 3, Nr 8, pp. 1914-1924 (USSR)

ABSTRACT: The interaction between the chlorides of rare earth metals and the chlorides of alkali and alkaline earth metals with their simultaneous crystallization from melts is of great scientific and practical interest. The interaction of the chlorides of lanthanum, cerium and neodymium, and the chlorides of potassium, cesium and magnesium in melts were investigated. The fusion diagrams of the following binary systems were constructed: $\text{LaCl}_3\text{-KCl}$, $\text{CeCl}_3\text{-KCl}$, $\text{NdCl}_3\text{-KCl}$, $\text{LaCl}_3\text{-CsCl}$, $\text{CeCl}_3\text{-CsCl}$, $\text{NdCl}_3\text{-CsCl}$.
The following types of compounds were found in the melts: M_3RCl_6 and K_2RCl_5 , in which R = rare earth element, and M = K, Rb, Cs. The stability of the compounds M_3RCl_6 is increasing

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SOV/78-3-8-32/48

The Interaction Between the Chlorides of Rare Earth Metals and the Chlorides of Alkali and Alkaline Earth Metals

from potassium to cesium and from lanthanum to neodymium. In the fusion diagram of the system $\text{CeCl}_3\text{-MgCl}_2$ there is a simple eutectic. The experiments showed that the chlorides of the rare earths form fusion diagrams with simple eutectics with the chlorides of the alkaline earth elements. The phase diagram of the ternary system $\text{CeCl}_3\text{-MgCl}_2\text{-KCl}$ was constructed and the limits of the primary crystallization of $\text{CeCl}_3\text{-MgCl}_2\text{-KCl}$, K_2CeCl_5 , K_3CeCl_6 , $\text{KCl} \cdot \text{MgCl}_2$ and $2 \text{KCl} \cdot \text{MgCl}_2$ were determined. There are 12 figures, 9 tables, and 8 references, 3 of which are Soviet.

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M. V. Lomonosova (Moscow Institute of Fine Chemical Technology imeni M. V. Lomonosov)

SUBMITTED: November 22, 1957

Card 2/2

AUTHORS: Marozov, I. G., Stefanyuk, S. L. SOV, 76-3-10-13 31
 TITLE: On the Kinetics of the Chlorination of Titanium Dioxide and Niobium Pentoxide (O kinetike khlorirovaniya tyutakol titana i pyatiodkisi niobiya)
 PERIODICAL: Zhurnal neorganicheskoy khimii, 1980, Vol 3, Nr 10, pp 2366-2374 (USSR)
 ABSTRACT: An investigation was carried out of the kinetics of the chlorination of titanium dioxide and niobium pentoxide in a mixture with carbon. In the reaction of gaseous chlorine with these oxides in the presence of solid reducing agents, the influence exerted by the partial pressure of chlorine, flow velocity, temperature of reaction, grain size of the initial materials and the type of the reducing agents were taken into account. A detailed description was given of an apparatus for the investigation of the kinetics of chlorination. The chlorination-reactor of the apparatus consists of quartz and is installed in an electric furnace. The chlorination was carried out at 700°C by means of briquettes containing approximately 1% carbon. The figures 2-4 show the kinetic curves of the chlorination of titanium dioxide at 60, 70 and 80°C. The penetration
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SOV 1958-10-10-111

On the Kinetics of the Chlorination of Titanium Dioxide and Niobium Pentoxide

of the chlorination into the depth of the briquettes was taken into account. The depth of chlorination means the thickness of the briquette layers that is chlorinated. It follows from the results obtained that all briquettes are chlorinated in the same time to the same depth, no matter how large they are. The depth of chlorination as a function of time was investigated with titanium briquettes. The chlorination of a mixture of titanium dioxide and niobium pentoxide was investigated at 600°C, and the influence of the temperature upon the velocity of chlorination was determined. Furthermore the change of the depth of chlorination in niobium pentoxide briquettes was investigated. The chlorination reaction of titanium dioxide and niobium pentoxide takes place in the presence of carbon at a temperature of above 600°C in the diffusion region. The velocity of chlorination can be increased by accelerating the flow velocity of chlorine. The reaction of chlorination can be accelerated by increasing the surface of the briquettes. There are 13 figures, 7 tables, and 6 references, 1 of which are Soviet.

Card 2/2

SUBMITTED:

May 20, 1958

AUTHORS: Morozov, I. S., Korshunov, B. G. 20-119-3-36/65

TITLE: On the Problem of the Thermodynamics of the Chlorination of the Rare-Earth Metal Oxides by Gaseous Chlorine (K voprosam termodinamiki khlorirovaniya okislov redkozemel'nykh metallov gazoobraznym khlorom)

PERIODICAL: Doklady Akademii Nauk SSSR, 1958, Vol. 119, Nr 3, pp. 523-525 (USSR)

ABSTRACT: This work is a continuation of the investigations by the authors on the thermodynamics and on the chemistry of the interaction of the rare-earth metal oxides with gaseous chlorine (reference 1,2). The equilibrium of the reactions of the type $4\text{MeCl}_3(\text{solid}) + 3\text{O}_2(\text{gaseous}) \rightleftharpoons 2\text{Me}_2\text{O}_3(\text{solid}) + 6\text{Cl}_2(\text{gaseous})$ was investigated, whereby Me denotes Sc, La, Nd, or Sm. The investigations were made on the conditions of the two-sided equilibrium, i.e. from the side chloride-oxygen and from the side oxide-chlorine. The preparations for the production of the metallic chlorides were placed to disposal by I. N. Zaozerskiy. The equilibrium of the gases was determined by the statistical method. The analysis took place in a thermostat glass buret. In the investigation of the equi-

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On the Problem of the Thermodynamics of the Chlorination of 20-119-3-36/65
the Rare-Earth Metal Oxides by Gaseous Chlorine

librium from the side oxide-chlorine a method, described by V. I. Smirnov and A. I. Tikhonov (Ref 4) was applied. The equilibrium in the system chloride-oxygen was, according to the experimental temperature, reached after 3-5 hours. The chlorination of the oxides and the oxidation of the metallic chlorides takes place without production of intermediates. Thus in the analysis of the oxides Sc, La, Nd, and Sm chlorinate at 400°C of chlorides are observed in the products, which form on that occasion of the composition ScCl_3 , LaCl_3 , NdCl_3 and SmCl_3 . The formulae, used for the computation of the equilibrium constants of the reactions $4 \text{MeCl}_3(\text{solid}) + 3 \text{O}_2(\text{gaseous}) \rightleftharpoons 2 \text{Me}_2\text{O}_3(\text{solid}) + 6 \text{Cl}_2(\text{gaseous})$, are written down. The here computed changes of the free energy of the reaction are composed in a table. The mean values of the heat values of the reactions in the mentioned temperature ranges are +24580; +25230; +25700; +25520 calories. A diagram illustrates the composition of the gaseous phase, which corresponds to the equilibrium, and another diagram illustrates the dependence of the logarithm $\lg K_r$ of the constant K_r of the reaction equilibrium on the reciprocal $1/T$ of the temperature T .

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On the Problem of the Thermodynamics of the Chlorination of 20-119-3-36/65
the Rare-Earth Metal Oxides by Gaseous Chlorine

The values of $\lg K_r = f(1/T)$ sufficiently exactly are on straight lines. According to the results, which were obtained here, the similar physical and chemical properties condition the resemblances of the thermodynamic properties.

There are 2 figures, 1 table, and 4 references, which are Soviet.

ASSOCIATION: Institut obshchey i neorganicheskoy khimii im. N. S. Kurnakova
(Institute of General and Inorganic Chemistry imeni N.S. Kurnakov)

PRESENTED: November 16, 1957, by I. I. Chernyayev, Member, Academy of Sciences, USSR

SUBMITTED: November 11, 1957

AVAILABLE: Library of Congress

Card 3/3

SCV/454-2-1-3/32

5(4)
AUTHORS: Morozov, I. S., Karanukov, B. G., Kozlov, V. V., Iva, V. V., I.

TITLE: Thermal and Tensimetical Investigation of the System $\text{NbCl}_5\text{-FeCl}_3\text{-NaCl}$

PERIODICAL: Izvestiya vysshikh uchebnykh zavedeniy. Khimiya i khimicheskaya tekhnologiya, 1959, Vol 2, Nr 4, pp 485 - 489 (USSR)

ABSTRACT: The investigation of the subject mentioned in the title is of interest with regard to the preparation of easily fusible melts containing niobium as well as to the purification of NbCl_5 from FeCl_3 . The system mentioned in the title is part of the quaternary system $\text{NbCl}_5\text{-FeCl}_3\text{-AlCl}_3\text{-NaCl}$. A thorough investigation of the latter will make it possible to produce melts with a crystallization temperature lower than that of the adjacent ternary systems (Ref 2). In the treatment of raw material containing niobium by chlorine a simpler condensation device is sufficient for easily fusible melts. The binary lateral systems adjacent to the system mentioned in the title have already been investigated earlier (Refs 3-5). In order to investigate the ternary system, five inner sections were made, and several mixtures determined

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Thermal and Tensimetric Investigation of the System NbCl_5 - FeCl_3 - NaCl SOV, 1961, 11, 1112

which do not form independent sections. Tables 1 and 2 show the results. The crystallization of the melts the figurative points of which are in the triangle NbCl_5 - FeCl_3 - NaCl in the phase diagram is concluded in the triple eutectic point E; the solid alloys consist of the phases NbCl_5 , FeCl_3 , and NaFeCl_4 . The tensimetric analysis was carried out in the system mentioned in the title was supposed to prove the results of the thermal analysis mentioned above. Moreover, the possibility of separating niobium chloride and iron chloride was to be examined. For this purpose, the vapor tensions over the mixtures of NbCl_5 , FeCl_3 , and NaCl were determined between 130 and 320°. For method and apparatus see reference 3. A table (without number) shows the composition of these mixtures in mol%. The results are shown in table 1 and figure 3. The results of the thermal analysis were proved by tensimetric investigations of the system mentioned in the title. Moreover, the possibility of separating niobium chloride and iron chloride by means of fractional distillation in the presence of NaCl was proved. In addition, vessels by Stepanov were mentioned in the paper.

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Thermal and Tensimetric Investigation of the System $\text{NbCl}_5\text{-FeCl}_3\text{-NaCl}$ SU 155-3-4-1 '58

There are 3 figures, 1 table, and 6 references, 5 of which are Soviet.

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni M. V. Lomonosova, Kafedra tekhnologii redkikh i rassseyanykh elementov (Moscow Institute of Fine Chemical Technology imeni M. V. Lomonosov, Chair of Technology of Rare and Dispersed Elements)

SUBMITTED: April 28, 1958

Card 1/1

SOV/78-4-2-38/40

5(2)

AUTHORS:

Sung-Yin-Chu . Morozov, I. S.

TITLE:

The Vapor Pressure of Hafnium Tetrachloride (Uprugost' para chetyrekhkhlorigo hafniya)

PERIODICAL:

Zhurnal neorganicheskoy khimii, 1959, Vol 4, Nr 4, p 492 (USSR)

ABSTRACT:

Anhydrous hafnium tetrachloride was produced by chlorinating metallic hafnium by chlorine gas. The melting point of the product is at 432°. The steam tension was determined by the flowing method in chlorine gas atmosphere. The dependence of the steam tension of hafnium and zirconium tetrachloride on the temperature was investigated. The results are shown in table 1. The steam tension of hafnium tetrachloride pressure in the temperature range of 200-312° according to the following equation:

$$\log P = - \frac{5390}{T} + 12.00$$

The steam tensions of hafnium and zirconium tetrachloride were drawn in a semi-logarithmic coordinate system. The sublimation temperature calculated is 314°; the evaporation

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SOV/78-4-2-38.40

The Vapor Pressure of Hafnium Tetrachloride

heat in the temperature range mentioned above is 24.6 kJ/mol.
There are 2 figures, 1 table, and 1 reference.

SUBMITTED: September 1, 1958

Card 2/2

5(4)

AUTHORS:

Morozov, I. S., Sun Yin-chu.

SCV/78-4-3-29/11

TITLE:

Investigation of the Phase Diagram of the Systems $ZrCl_4$ -KCl, $ZrCl_4$ -CsCl, $HfCl_4$ -NaCl, $HfCl_4$ -KCl, $HfCl_4$ -CsCl (Izuchenie diagramm sostoyaniya sistem $ZrCl_4$ -KCl, $ZrCl_4$ -CsCl, $HfCl_4$ -NaCl, $HfCl_4$ -KCl, $HfCl_4$ -CsCl)

PERIODICAL:

Zhurnal neorganicheskoy khimii, 1959, Vol 4, Nr 3, pp 678-683 (USSR)

ABSTRACT:

By thermal analysis the interaction of the chlorides of zirconium and hafnium with the chlorides of sodium, potassium, and cesium was investigated. In the system $ZrCl_4$ -KCl two eutectics occur at 24.2 mole % $ZrCl_4$ with a melting point 534° and at 57.8 mole % $ZrCl_4$ with the melting point 220° . In all melts with a zirconium content up to 33.3 mole % a thermal effect occurs which is probably caused by the decomposition of K_2ZrCl_6 . In the system $ZrCl_4$ -CsCl a stable chemical compound Cs_2ZrCl_6 forms with the melting point at 305° . The eutectic formed by Cs_2ZrCl_6 and cesium chloride melts at 572° and

Card 1/4

Investigation of the Phase Diagram of the Systems SOV 72-1-1-100-101
 $ZrCl_4$ -KCl, $ZrCl_4$ -CsCl, $HfCl_4$ -NaCl, $HfCl_4$ -KCl, $HfCl_4$ -CsCl

contains 15.2 mole % $ZrCl_4$. The eutectic formed by Cs_2ZrCl_6 and zirconium chloride contains 67.2 mole % $ZrCl_4$ and melts at 286° . In the system $HfCl_4$ -NaCl the chemical compound Na_2HfCl_6 is formed with the melting point at 460° . Three polymorphic transformations take place in the compound at 384° , 440° , and 494° . Two eutectics occur on the melting diagram: at the melting point 510° with 26.6 mole % $HfCl_4$ and at the melting point 330° with 59.1 mole % $HfCl_4$. The phase diagram of the system NaCl- $HfCl_4$ in the range of $73-100^\circ$ and $HfCl_4$ was not found. In the system $HfCl_4$ -KCl the chemical compound K_2HfCl_6 is formed with a melting point at 302° . The phase diagram shows the following eutectics: at 60° formed by K_2HfCl_6 and potassium chloride with 22.4 mole % $HfCl_4$; at 212° formed by K_2HfCl_6 and $HfCl_4$ with 62 mole % $HfCl_4$; at 550° the

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Investigation of the Phase Diagram of the Systems SCV/78-1-3-28/31
 $ZrCl_4$ -KCl, $ZrCl_4$ -CsCl, $HfCl_4$ -NaCl, $HfCl_4$ -KCl, $HfCl_4$ -CsCl

thermal effect is probably caused by the decomposition of K_2HfCl_6 . In the system $HfCl_4$ -CsCl Cs_2HfCl_6 forms with the melting point at 820° . Two eutectics are formed at 540° by Cs_2HfCl_6 and cesium chloride with 18.1 mole % $HfCl_4$ and at 302° by Cs_2HfCl_6 and $HfCl_4$ with 65.1 mole % $HfCl_4$. Based upon these results the phase diagrams of the systems were plotted and are given by figures 1, 2, 3, 4, and 5. In all systems compounds of the type: $Me_2^{I,IV}Cl_6$ (Me^I -Na, K, Cs; Me^{IV} -Zr, Hf) are formed. Compound Cs_2ZrCl_6 has a polymorphic transformation at 377 and 311° and compound Na_2HfCl_6 at 484 , 440 , and 394° . The compounds Cs_2ZrCl_6 at 805° and Cs_2HfCl_6 at 820° have the highest melting points. There are 5 figures, 6 tables, and 5 references, 2 of which are Soviet.

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Investigation of the Phase Diagram of the Systems SOV/73-4-3-28/31
 $ZrCl_4$ -KCl, $ZrCl_4$ -CsCl, $HfCl_4$ -NaCl, $HfCl_4$ -KCl, $HfCl_4$ -CaCl

SUBMITTED: September 1, 1959

Card A/1

5(4)

SOV/78-4-6-41/44

AUTHORS: Morozov, I. S., Ionov, V. I., Korshunov, B. G.

TITLE: Thermal Analysis of the System $\text{NdCl}_3\text{-MgCl}_2\text{-KCl}$ (Termicheskiy analiz sistemy $\text{NdCl}_3\text{-MgCl}_2\text{-KCl}$)

PERIODICAL: Zhurnal neorganicheskoy khimii, 1959, Vol 4, Nr 6, pp 1457 - 1458 (USSR)

ABSTRACT: The system $\text{NdCl}_3\text{-MgCl}_2\text{-KCl}$ was investigated by the method of the thermal analysis; the phase diagram was constructed and is shown in figure 1. 7 internal sections were investigated in the three-component system. It was found that 7 regions of primary crystallization are formed on the surface of the liquidus corresponding to the compounds KCl , NdCl_3 , MgCl_2 , K_3NdCl_6 , K_2NdCl_6 , KCl.MgCl_2 and 2KCl.MgCl_2 . The four-phase equilibrium in the system $\text{NdCl}_3\text{-MgCl}_2\text{-KCl}$ is given in a table. The existence of the compound 2KCl.MgCl_2 was confirmed in the system $\text{MgCl}_2\text{-KCl}$. There are 1 figure, 1 table, and 6 references, 4 of which are Soviet.

Card 1/2

05873

SOV/78-4-11-26/50

5(2)

AUTHORS: Morozov, I S , Sung Yin-chu

TITLE: Temperature Stability of the Compounds of Zirconium- and Hafnium Chloride With the Chlorides of Alkali Metals

PERIODICAL: Zhurnal neorganicheskoy khimii, 1959, Vol 4, Nr 11, pp 2551 - 2553 (USSR)

ABSTRACT: At present, great efforts are being made to develop procedures for the preparation of metallic hafnium and zirconium by electrolytic treatment of their halogen compounds in chloride- and fluoride melts (Ref 1). Compounds of the composition Me_2ZrCl_6 and Me_2HfCl_6 (Me= Na, K, Rb, Cs) result from the melting of alkali chlorides. In order to explain the possibility of using alkali hexachlorozirconates and the corresponding hafnium compounds as electrolytes, the authors investigated the temperature stability of these compounds. They measured: vapor pressure of ZrCl_4 and HfCl_4 over the melt of the hexachlorine compounds (Tables 1,2), temperature dependence of the vapor pressure of the tetrachlorides over the melts of the hexachlorine compound (Tables 3,4) as well as the variation of free energy

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05873

Temperature Stability of the Compounds of Zirconium-
and Hafnium Chloride With the Chlorides of Alkali Metals

SOV/78-4-11-26/50

and entropy and the heat effect of thermal dissociation of the alkali hexazirconates and hafnates (Table 5). The hexachlorine compounds become more stable with decreasing ionic radius of the alkali metal: $\text{Cs}_2\text{ZrCl}_6 > \text{K}_2\text{ZrCl}_6 > \text{Na}_2\text{ZrCl}_6$; $\text{Cs}_2\text{HfCl}_6 > \text{K}_2\text{HfCl}_6 > \text{Na}_2\text{HfCl}_6$. Herefrom it follows that the hexachlorohafnates are somewhat more stable than the corresponding zirconium compounds. There are 5 tables and 6 references, 2 of which are Soviet.

SUBMITTED: September 3, 1958

Card 2/2

5.2200

307

AUTHORS: Yerdokimov, V. I., Morozov, I. S.

TITLE: Letter to the Editor. Synthesis of Stannous Chloride From Elements

PERIODICAL: Izvestiya Akademii nauk SSSR. Otdeleniye Khimicheskikh nauk, 1960, Nr 1, p 1-7 (USSR)

ABSTRACT: The authors report that stannous chloride was obtained for the first time by direct synthesis from the elements. A good contact between the gaseous chlorine and the reacting surface of tin is important. Two different methods were used: (1) The process was conducted at a temperature higher than the mp of SnCl_2 (100°), thus removing SnCl_2 formed by evaporation. (2) The process was conducted at a temperature slightly higher than mp of tin ($235-300^\circ$) thus by constant renovation of the reacting tin surface. The proposed methods are of industrial importance. They can be used for

Card 1/2

Letter to the Editor. Synthesis of
Stannous Chloride From Elements

18087
SOV/12-10-1-1

the preparation of other products. Anhydrous stannous
trichloride was obtained from tetra-chloride by the
proposed methods.

ASSOCIATION: N. S. Kurnakov Institute of General and Inorganic
Chemistry Academy of Sciences USSR (Institute of General
Inorganic Chemistry named N. S. Kurnakov
Akademii nauk SSSR)

SUBMITTED: October 20, 1959

Card 2/2

S/149/60/000/003/10/12/01
A006/A001

AUTHORS: Ionov, V.I., Korshunov, B.G., Kokorev, V.V., Morozov, I.S.

TITLE: Physical and Chemical Study on Interaction of Thorium Chloride
With Chlorides of Alkali-Metals and Cerium in Melts

PERIODICAL: Izvestiya vyssikh uchebnykh zavedeniy, Tsvetnaya metallurgiya,
1960, No. 3, pp. 102-108

TEXT: Literature data on thorium chloride chemistry are incomplete and obsolete. The authors investigated the interaction of thorium chloride with chlorides of sodium, potassium, cesium and cerium in melts, for the purpose of completing the knowledge about the physical and chemical nature of some technological processes of thorium chloride preparation. Thorium chloride was prepared by chlorination of thorium dioxide mixed with charcoal from sugar, by gaseous chlorine at 1,000-1,050°C. The melting temperature of the chloride obtained was 750°C. Vapor tension of thorium chloride corresponding to its melting temperature was about 80 mm Hg. Cerium chloride was prepared by the method described in Reference 16. The melting temperatures of chlorides of sodium, potassium, cesium and cerium were 800, 776, 646 and 802°C, respectively. The chloride systems

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S/149/60/000/003/010/012/001
A005/A001

Physical and Chemical Study on Interaction of Thorium Chloride With Chlorides of Alkali-Metals and Cerium in Melts

were studied by thermal and tensimetric analyses. The thermal analysis was made by differential and plain recording of heating and cooling curves on a N.S. P. nakov pyrometer. The temperature was measured with a platinum-chromium thermocouple graduated according to conventional datum points. Melting points of salt mixtures and recording of curves was made in quartz glass ampoules. After filling the container with the salt mixture, the air was evacuated by a hydrogen chloride current. The container was then sealed and placed in a furnace. The cooling rate was 40-100°C per minute, depending on the temperature range. It was established that in the ThCl_4 -MeCl systems the components form ternary compounds of the MeThCl_5 , Me_2ThCl_6 , Me_3ThCl_7 type (excepted the ThCl_4 -NaCl system). The NaThCl_5 system melts incongruently at 370°C, KThCl_5 and CsThCl_5 melt incongruently at 428 and 490°C respectively. Na_2ThCl_6 melts congruently at 360°C, K_2ThCl_6 and Cs_2ThCl_6 melt incongruently at 406 and 573°C respectively. K_3ThCl_7 and Cs_3ThCl_7 melt congruently at 705 and 720°C respectively. Data obtained by thermal analysis are confirmed by tensimetric investigation of the system. The tensimetric analysis was made by the dynamic method. The determination of chloride vapor tension was conducted in chlorine atmosphere. The amount of the initial

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3/149/60/000/003/010/012/01
A006/A001

Physical and Chemical Study on Interaction of Thorium Chloride With Chlorides of Alkali Metals and Cerium in Melts

mixture in all experiments was about 30 g. The salt mixtures were melted in sealed ampoules cooled, crushed in argon atmosphere, and placed into the apparatus. The amount of chlorine passed was determined from the increase in weight of the potash bulbs filled with 25% NaOH solution. The rate of the chlorine current was sufficient to saturate the volatile chlorides. The quantity and composition of the sublimate were determined by chemical analysis and the pressure in the apparatus by the sum of atmospheric and excess pressure obtained when the gas passed through the absorption flasks. A formula is given to calculate the partial vapor tension of the mixture components, and values of vapor tension of thorium chloride over Na_2ThCl_6 , K_3ThCl_7 and Cs_3ThCl_7 at various temperatures are given. It was established that the thermal stability of thorium chloride combined with alkali metal chlorides changed regularly, increasing from sodium chloride to cesium chloride. The method of thermal analysis was used to study feasibility of the systems $\text{ThCl}_4 - \text{CeCl}_3$ and $\text{ThCl}_4 - \text{CeCl}_3 - \text{NaCl}$, which was shown in feasibility

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S/149/60/000/003/010/012/001
A006/A001

Physical and Chemical Study on Interaction of Thorium Chloride With Chlorides of Alkali-Metals and Cerium in Melts

diagrams. There are 4 figures, 1 table and 20 references: 5 Soviet, 8 English, 3 French and 4 German.

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii (Moscow Institute of Fine Chemical Technology). Kafedra khimii i tekhnologii redkikh i rasseyannykh elementov (Department of the Chemistry and Technology of Rare and Dispersed Elements)

SUBMITTED: July 1, 1959

Card 4/4

SHEVTSOVA, Z.N.; MOROZOV, I.S.; YEFREMOVA, O.A.

Fusibility diagram for the system praseodymium chloride-
magnesium chloride - potassium chloride. Izv. vys. ucheb. zav.;
tsvet. met. 3 no.3:109-111 '60. (MIRA 14:3)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii, Kafedra
khimii i tekhnologii redkikh i rasseyannykh elementov.
(Praseodymium chloride--Electrometallurgy)
(Melting points)

Study of the Interactions of the Chlorides of the Rare Earths With the Chlorides of the Alkaline Earth and Alkali Metals in Molten Thermal Analysis of the System $\text{CaCl}_2 - \text{NaCl} - \text{BaCl}_2$

Abstract: The authors have studied the system $\text{CaCl}_2 - \text{NaCl} - \text{BaCl}_2$ in the range of composition from 0 to 100% CaCl_2 . On the basis of their results, the authors have determined the crystallization fields of CaCl_2 , $\text{CaCl}_2 \cdot \text{NaCl}$, and NaCl in the liquid phase. The authors have also determined the phase fusion diagram of the system. The liquid phase consists of CaCl_2 (13.4%), $\text{CaCl}_2 \cdot \text{NaCl}$ (36.8%), NaCl (47.0%), and BaCl_2 (1.8%) at 1100°C. The crystalline CaCl_2 , $\text{CaCl}_2 \cdot \text{NaCl}$, and NaCl contain 13.4%, 36.8%, and 47.0% CaCl_2 , respectively, and melt at 1010, 1010, and 1010°C, respectively. There are no eutectics, peritectics, and congruent melting points in the system.

Car: 1/4

S/53/60/007/007/007/007/007
10-1/2000

Thermal Analysis of the System PbCl_2 - PbO - NaCl

ASSOCIATION. Moskovskiy institut tankoy khimicheskoy tekhnologii im. M. V. Lomonosova; Katedra tekhnologii mekhanicheskogo rasseyaniya elementov (Moscow Institute of Fine Chemical Technology ineni M. V. Lomonosov; Chair of Technology of Rare and Disperse Elements).

SUBMITTED: November 5, 1950

Fig. 1.

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S-149/60/000/005/004/015
A006/A001

AUTHORS Korsunov, V.G., Morozov, I.S., Ikonov, V.I., and Zorina, M.A.
TITLE Physical and Chemical Studies of the $\text{AlCl}_3\text{-FeCl}_3\text{-NaCl}$ System
PERIODICAL Izvestiya vysshikh shkolnykh zavedeniy. Tsvetnaya metallurgiya, 1960, No. 5, pp. 67-71

TEXT The authors studied the interaction of aluminum, iron and sodium chlorides by the method of thermal and tensimetric analysis for the purpose of developing chemical and physical bases for the refining of chlorides of titanium and other metals. The necessary aluminum and iron chlorides were obtained by chlorination with gaseous chlorine of the respective metals, sodium chloride was preliminary remelted. Melting temperatures of the chloride salts of aluminum, iron and sodium were 194, 303 and 800°C respectively. Due to the fact that aluminum and iron chlorides have high vapor tensions at their melting temperatures, different mixtures of the system were melted in molybdenum or quartz glass Stepanov containers. The thermal analysis of the system was made by recording the cooling curves on a N.S. Zhurav type pyrometer. The temperature was measured with a nichrome-constantan thermocouple graduated according to GOST 1-5.

S 149/60 000/005 00- 015
A006/A001

Physical and Chemical Studies of the AlCl_3 - FeCl_3 - NaCl System

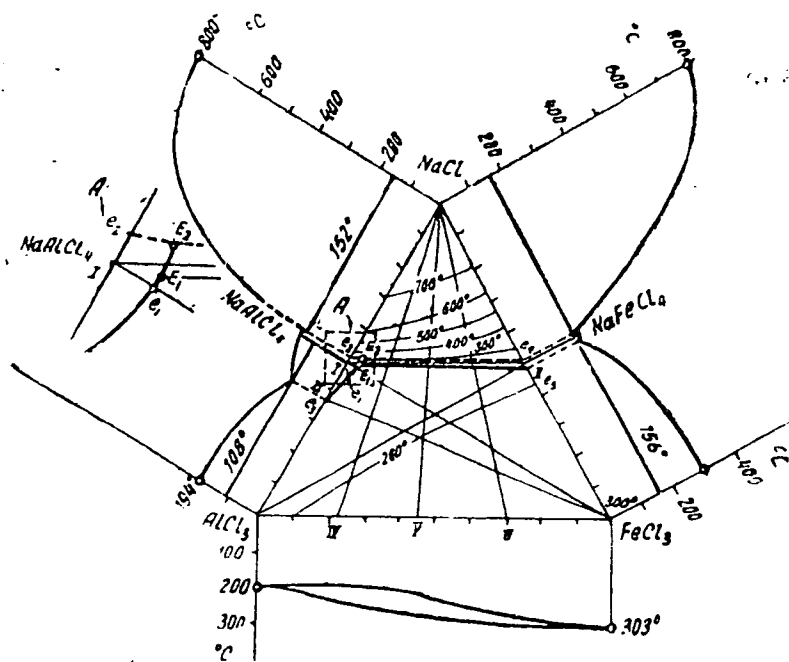
ventional datum points. The ternary system¹ was studied by investigating six internal sections (Figure 1), whose direction was mainly determined by the location of non-variable equilibrium points on the lateral binary diagrams. The compositions are expressed in mole percent. The thermometric analysis was made to confirm the results of the thermal analysis of the system and to investigate the vapor tension of NaAlCl_4 and NaFeCl_4 compounds during their joint presence under conditions of sodium chloride excess. Vapor tension was determined in chlorine atmosphere by the dynamic method. The formation of a NaFeCl_4 compound in the FeCl_3 - NaCl system and its vapor tension were determined. The results of thermometric analysis are given in a table. The feasibility diagram plotted may be used for calculations connected with the extraction of chlorides of titanium and other elements from aluminum and iron compounds by means of sodium chloride

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S/149/60/000/005/004/015
A006/A001

Fig. 1

Fig. 1
Fusibility diagram
of the AlCl_3 - FeCl_3 - NaCl
System



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A006/A001

Physical and Chemical Studies of the AlCl_3 - FeCl_3 - NaCl System

Results of the tensiometric analysis of three mixtures of the AlCl_3 - FeCl_3 - NaCl system

No. No. of mixtures	Temperature, °C	Vapor tension, mm Hg	
		NaAlCl_4	NaFeCl_4
1	500	0,0	0,0
	530	0,2	2,2
	586	1,2	11,8
	620	2,6	15,9
	650	5,1	21,4
	670	6,7	25,0
2	362	0,0	0,0
	423	0,9	2,2
	477	1,2	5,9
	558	3,5	12,9
	590	4,7	21,1

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S 149/60/000 000/000-000
ACC6 1A001

Physical and Chemical Studies of the Al_2Cl_3 - $FeCl_3$ - $NaCl$ System

No. of mixtures	Temperature °C	Vapor tension, mm Hg	
		Al_2Cl_3	Fe_2Cl_6
3	150	32,0	1.1
	161	67.2	3.3
	173	129,0	4.9
	184	272,8	6.1

There are 2 figures, 1 table and 22 references 12 Soviet, 6 English, 2 French and 2 German.

ASSOCIATIONS Moskovskiy institut finykh khimicheskoy tekhnologii (Moscow Institute of Fine Chemical Technology), Kafedra khimii i tekhnologii redkikh i rasseyannykh elementov (Department of Chemistry and Technology of Rare and Dispersed Elements)

SUBMITTED October 27, 1959

Card 5/5

57598/60/000-001/011/000
0217/0302

AUTHORS Morozov, I.S. and Toptygin, D.Ya
TITLE Physico-chemical basis of purification processes for titanium tetrachloride
SOURCE Akademiya nauk SSSR, Institut metallurgii. Titan i yego splavy. No. 4. Moscow, 1960. Metallurgiya titana, 102-114

TEXT The study of systems formed by the chlorides of Nb, Ta, Al, Fe and V with $TiCl_4$ was carried out by three methods. Thermal analysis, solubility and tensile testing. Owing to the tendency of the above chlorides to hydrolyze and volatilize, the thermal analysis of the mixtures was carried out in sealed containers. The chloride mixtures (2-5 g) were melted in a furnace, and, in order to ensure even cooling, were each placed in a porcelain container lined with asbestos wool. A thermocouple was used to plot cooling curves and in individual cases, a differential thermal analysis of the mixtures was carried out. Ensuring uniform cooling of the mixtures from 300 C to the temperature at which the

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S/598/60/000/004/011/020
D217/D302

Physico-chemical basis ...

eutectic solidifies at -24°C : was difficult, and, therefore, the latter was determined separately. For this purpose, the chlorides in their containers were cooled by a mixture of dry ice and alcohol, and heating curves were recorded by means of a thermocouple. Investigation by the solubility method was carried out between 18 and 110°C . The mixture of chlorides was placed in a container consisting of a test tube with a side branch. The container with the chlorides was sealed, heated until the chlorides were completely molten and placed in a thermostat maintained at a definite temperature for 20-40 hours. After equilibrium had established itself between the solid phase formed and the solution, part of the transparent solution was decanted into the side branch. The latter was cut off and the composition of the liquid phase was determined by chemical analysis. On distilling TiCl_4 from the solution containing excess solid sodium chloride, no aluminum chloride could be detected in the distillate. This property was taken advantage of in the analysis for the separation of the main body of TiCl_4 from AlCl_3 . A 5-10 g probe was transferred to a Wurtz flask and 1-2 g dry NaCl added. TiCl_4 was

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D217/D302

Physico-chemical basis ...

distilled and the residue dissolved in dilute HCl. From the solution obtained, aluminum was precipitated with NaF as cryolite or by ammonia as the hydroxide. Weighing was carried out in the form of Al_2O_3 which always contained some titanium dioxide. Ti was determined calorimetrically by the reaction with H_2O_2 . Ta and Nb were separated similarly from $TiCl_4$, however in this case small traces of Ta and Nb chlorides were detected in the distillate. Final separation of Ta and Nb from Ti was carried out by means of phenyl arsenic acid. A visual method was used to study mixtures of $TiCl_4$ and $FeCl_3$ containing 0.5-2% $FeCl_3$. It was found that $TiCl_4$ can be purified from $AlCl_3$ and $FeCl_3$ by means of alkali metal chlorides. The formation of the chemical compound $NaFeCl_4$ in the system $FeCl_3$ -NaCl was proved. Separation of Ta and Nb in the form of chlorides during dissolution of a mixture of the latter in $TiCl_4$ was found to be impossible. The degree of solubility of the chlorides of

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D217/D302

Physico-chemical basis ...

Ta, Nb, Al and Fe in $TiCl_4$ was found to vary considerably in the presence of other chlorides. The study of systems containing niobium pentachloride by a visual method is difficult owing to the presence of traces of niobium perchlorate. The solubility of $NbOCl_3$ at low temperatures is extremely low. At 280-300°C transparent solutions of up to 2% $NbOCl_3$ content were obtained. $TiCl_4$ does not react with the compounds NH_4AlCl_4 and NH_4TaCl_6 . On heating the above chlorides with $TiCl_4$, stratification occurs in a wide concentration interval. The compound NH_4NbCl_6 is decomposed by $TiCl_4$. There are 16 figures, 7 tables and 12 references: 3 Soviet-bloc and 7 non-Soviet bloc. The references to the 4 most recent English-language publications read as follows: S.L. May, H.W. Henderson and H.A. Johansen. Ind. Eng. Chem, 46 (12), 2495, 1954; I. N. Rowe and E.R. Opie. J. of Met., 7, 11, 1955, 1183; H.F. Johnston, H. Weihgarther and W.E. Winsche. J. Am. Chem. Soc., 64, 241, 1942; P. Fireman. J.M. Soc., 26, 745, 1904.

Card 4/4

5.2100
5.2200(E)

5-2)

AUTHORS: Morozov, I. S., Toptygin, D. Ya.

68111
SOV/78-5-1-15/45

TITLE: Thermal Stability of Hexachlorotitanates of Monovalent Metals

PERIODICAL: Zhurnal neorganicheskoy khimii, 1960, Vol 5, Nr 1, pp 88 - 90 (USSR)

ABSTRACT: The possibility of obtaining pure titanium by the electrolysis of titanium halide compounds in a melt of alkali chlorides or alkali fluorides induced the authors to investigate the thermal stability of the complex compounds forming in this connection. They investigated the reaction between $TiCl_4$ and alkali chlorides, by directing $TiCl_4$ -vapor-saturated chlorine over heated alkali chloride. $CsCl$ was completely transformed into Cs_2TiCl_6 at $660 - 700^\circ$. In the case of $RbCl$ the reaction product always still contains residues of unreacted $RbCl$. KCl does not form any complex salt at $600 - 700^\circ$, and 10% of K_2TiCl_6 at most were obtained at $400 - 500^\circ$. $NaCl$ did not react with $TiCl_4$. The vapor pressure of $TiCl_4$ was determined on specially prepared

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Thermal Stability of Hexachlorotitanates of Monovalent Metals 68111
SOV/78-5-1-15/45

pure compounds Me_2TiCl_6 ($\text{Me} = \text{Cs}, \text{Rb}, \text{K}, \text{Tl}$) (Table 1, Figs 1,2). This investigation confirmed as well that Cs_2TiCl_6 exhibits the highest thermal stability. This decreases in the series $\text{Cs}_2\text{TiCl}_6 > \text{Rb}_2\text{TiCl}_6 > \text{K}_2\text{TiCl}_6 > \text{Tl}_2\text{TiCl}_6$. Table 2 supplies the calculated decomposition temperatures of chlorotitanates of Cs, Rb, K, Tl. Despite the large differences in the decomposition temperatures the calculated decomposition reaction heats amount to about 33 kcal/mol with all four chlorotitanates. Table 3 shows the change of free energy in the thermal dissociation of alkali chlorotitanates. There are 2 figures, 3 tables, and 5 references.

SUBMITTED: September 10, 1958

Card 2/2

S/078/60/005/06/09/030
B004/B014

AUTHORS: Ionov, V. I., Morozov, I. S., Korshunov, B. G.

TITLE: Thermal Analysis of the Systems NdCl_3 - FeCl_2
 FeCl_2 - NaCl , FeCl_2 - KCl , FeCl_2 - CsCl and
 NdCl_3 - FeCl_2 - KCl

PERIODICAL: Zhurnal neorganicheskoy khimii, 1960, Vol. 5, No. 6
pp. 1248 - 1253

TEXT: The authors specify the following data obtained from experiments. /B
melting-point diagram of the system NdCl_3 - FeCl_2 with a eutectic at
608°C and 59.8 mole % FeCl_2 (Fig. 1); melting-point diagram of the system
 FeCl_2 - NaCl with a eutectic at 370°C and 44 mole % FeCl_2 (Fig. 2); mel-
ing-point diagram of the system FeCl_2 - KCl (Fig. 3). The compounds
 KFeCl_3 (α - and β -modification) and K_2FeCl_4 are formed in the latter. The

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Thermal Analysis of the Systems $\text{NdCl}_3 - \text{FeCl}_2$, S/078/60/005/06/09/030
 $\text{FeCl}_2 - \text{NaCl}$, $\text{FeCl}_2 - \text{KCl}$, $\text{FeCl}_2 - \text{CsCl}$, and B004/B014
 $\text{NdCl}_3 - \text{FeCl}_2 - \text{KCl}$

eutectic $\text{KFeCl}_3 + \text{K}_2\text{FeCl}_4$ melts at 340°C and corresponds to a content of 39.8 mole % FeCl_2 . The eutectic $\text{KFeCl}_3 + \text{FeCl}_2$ melts at 380°C with a content of 52.2 mole % FeCl_2 . In the system $\text{FeCl}_2 - \text{CsCl}$ (Fig. 4), the compounds CsFeCl_3 and Cs_2FeCl_4 arise with the eutectics $\text{Cs}_2\text{FeCl}_4 + \text{CsCl}$ (508°, 21.4 mole % FeCl_2), $\text{CsFeCl}_4 + \text{CsFeCl}_3$ (522°, 38.0 mole % FeCl_2) and $\text{CsFeCl}_3 + \text{FeCl}_2$ (498°, 69.3 mole % FeCl_2). Hence, the thermal stability of compounds of FeCl_2 with alkali chlorides increases from Na to Cs. Eight sections were examined in the system $\text{NdCl}_3 - \text{FeCl}_2 - \text{KCl}$ (Figs. 5-12).

The melting-point diagrammatically shown in Fig. 12 was constructed on the strength of these results. Numerous conversions were detected below the liquidus surface. Data on the four ternary eutectic points and one

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Thermal Analysis of the Systems NdCl_3 - FeCl_2 S/078/60/005/06/09/030
 FeCl_2 - NaCl , FeCl_2 - KCl , FeCl_2 - CsCl and B004/B014
 NdCl_3 - FeCl_2 - KCl ✓B

ternary peritectic point are supplied. There are 2 figures and
4 references: 2 Soviet and 2 American.

Card 3/3

MOROZOV, I.S.; TOPTYGINA, G.M.

Solubility in the system $\text{TiCl}_3 - \text{NH}_4\text{Cl} - \text{H}_2\text{O}$ (0° isotherm).

Zhur.neorg.khim. 5 no.7:1637-1638 J1 '60.

(MIRA 13:7)

(Titanium chloride) (Ammonium chloride)

S/078/60/005/011/011/025
B015/B060

AUTHORS: Morozov, I. S. Topygina, G. M.

TITLE: Chloro Titanates and Chloroxy Titanates of Alkali Metals and Ammonium

PERIODICAL: Zhurnal neorganicheskoy khimii, 1960, Vol. 5, No. 11, pp. 2518-2529

TEXT: The authors studied the solubility in systems consisting of hexachloro titanates of cesium, rubidium, ammonium, and potassium with hydrogen chloride and water, since these are used in technological processes. The solubility of cesium- and rubidium hexachloro titanates was studied in the concentration range from 0 to 44.5% HCl at 0°C (Tables 1-2, results), and maximum solubility was established in hydrochloric acid Cs_2TiCl_6 - 0.44% TiO_2 and Rb_2TiCl_6 - 0.96% TiO_2 . At an HCl concentration of 28.6% for cesium and 30.0% for rubidium there forms a solid phase corresponding to compound $\text{Me}_2\text{TiOCl}_4 \cdot \text{H}_2\text{O}$ (Table 3), whose composition was confirmed by means of Schreinemakers' method. If there is

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Chloro Titanates and Chloroxy Titanates
of Alkali Metals and Ammonium

S/078/60/005/011/013/025
B015/BC60

an excess of alkali metal chloride in the solution, the solubility of the complex salt drops, without there resulting another compound (Table 4). The solubility of ammonium hexachloro titanate $(\text{NH}_4)_2\text{TiCl}_6$ was studied in the concentration range from 17 to 44.5% HCl at 0°C (Table 5, results), and, in agreement with data by Seidel and Fischer (Ref. 10), 0.12% TiO_2 was found as the minimum solubility. The maximum solubility is 1.16% TiO_2 (35.15% HCl). A compound of composition $3\text{NH}_4\text{Cl} \cdot 2\text{TiOCl}_2 \cdot 4\text{H}_2\text{O}$ (Table 6) was identified and its solubility was determined (Table 7). The minimum solubility of K_2TiCl_6 in hydrochloric acid saturated with HCl is 0.06% TiO_2 at 0°C (Table 8). Unlike the abovementioned chloro titanates, potassium does not form any chloroxy titanate crystal hydrate. The separating solid phases were subjected to a thermal analysis, and heating curves were drawn for 20° to 1000°C in argon or air. For cesium hexachloro titanate two endothermal effects were observed at a 650°C melting point and a dissociation at 685°C. Rb_2TiCl_6 has only an endothermal effect at 658°C (the melting point) and simultaneous dissociation. Ammonium- and potassium

Card 2/3

Chloro Titanates and Chloroxy Titanates
of Alkali Metals and Ammonium

S/078/60/005/011/C13/025
BO15/BO60

hexachloro titanate contain 1 mole of crystal water, so that there appears the corresponding endoeffect. The chloroxy titanate compounds obtained were examined by crystal-optical analysis, and the refractive indices (Table 9) were determined. On the basis of these new compounds, several aspects of the state of tetravalent titanium in hydrochloric acid solutions are discussed. An equilibrium is assumed in diluted hydrochloric acid

solutions: $[\text{Ti}(\text{OH})_2]^{2+} \rightleftharpoons \text{TiO}^{2+} + \text{H}_2\text{O}$, while a complex anion results with

rising acid concentration: $[\text{Ti}(\text{OH})_2]^{2+} + 4 \text{Cl}^- \rightleftharpoons [\text{Ti}(\text{OH})_2\text{Cl}_4]^{2-}$. Yellow-colored hexachloro titanate anion $[\text{Ti}(\text{OH})_2\text{Cl}_4]^{2-} + 2\text{Cl}^- + 2\text{H}^+ \rightleftharpoons [\text{TiCl}_6]^{2-}$ ✓

+ $2\text{H}_2\text{O}$ forms in concentrated hydrochloric acid solutions. The complete hydrolysis in poorly acid solutions runs as follows: $[\text{Ti}(\text{OH})_2]^{2+}$

+ $2\text{H}_2\text{O} \rightleftharpoons \text{Ti}(\text{OH})_4 + 2\text{H}^+$, which does not, however, exclude the formation of intermediate stages. There are 7 figures, 9 tables, and 15 references: 5 Soviet and 8 German.

Card 3/3

S/080/60/033/008/001/013
A003/A001

AUTHOR: Morozov, I.S.

TITLE: The Chlorine Method in the Industry of Rare and Non-Ferrous Metals

PERIODICAL: Zhurnal prikladnoy khimii, 1960, Vol. 33, No. 8, pp. 1685-1700

TEXT: The application of gaseous chlorine in metallurgy is very promising. Its application in the metallurgy of rare metals and non-ferrous metals was investigated by G.G. Urazov, I.S. Morozov, V.I. Spitsyn, N.P. Sazhin, D.M. Chizhikov, et al. In the treatment of titano-niobates of rare-earth metals and titanium-containing slags the ore concentrate is mixed with coke and treated with gaseous chlorine at a high temperature. In this case the chlorine consumption is 85% and the degree of extraction 92-100%. In the chlorination of a pyrochlore concentrate with a Nb₂O₅ content of 6.5% at a temperature of 600°C and a chlorine supply rate of 1.12 g/min the process lasts 30-40 min. The degree of extraction is 99%. In the reaction $\text{MeO}_2 + 2\text{Cl}_2 \rightleftharpoons \text{MeCl}_4 + \text{O}_2$ the equilibrium is shifted to the right side, if the initial metal oxide under the conditions of chlorination is less stable than the chloride. If it is more stable, the free oxygen must be bound by carbon or carbon monoxide. The chlorination of niobium pentoxide was carried out at 700,

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S/080/60/033/508/001/013
AC03/A001

The Chlorine Method in the Industry of Rare and Non-Ferrous Metals

800, 900, 1,000 and 1,100°C and at a rate of the chlorine flow of up to 70 cm³/min. At 1,100°C the chlorination of Nb₂O₅ reached 0.2490 g/l Cl₂, the chlorine consumption 6.30%. In the chlorination of zirconium dioxide, carried out at 1,000, 1,100 and 1,200°C, ZrO₂ is chlorinated at 1,200°C at the rate of 0.0248 g/l Cl₂. The equilibrium of reactions of the type 4MeCl₃ (solid) + 3O₂ (gaseous) ⇌ 2Me₂O₃ (solid) + 6Cl₂ (gaseous), where Me is ⁹⁰Zr, ⁹¹Zr, ¹⁴⁰Nd or ¹⁵²Sm, was investigated. The equilibrium was reached in the chloride-oxygen system after 3-5 days depending on the temperature. The presence of a reducing agent, e.g., carbon, reduces the chlorination temperature of Ta₂O₅ from 1,200 to 300°C. Bardin and Reznichenko (Ref. 12) found that the chlorination of TiO₂ in the presence of carbon monoxide proceeds intensively at 1,000-1,050°C. The kinetics of the chlorination of briquets made of a metal oxide and carbon was studied. It was shown that at temperatures of up to 520°C the reaction rate increases with the temperature and does not depend on the chlorine flow rate. Above 600°C the chlorination of zirconium and titanium dioxides and niobium pentoxide proceeds in the diffusion region and increases with the chlorine flow rate. The separation of niobium and tantalum from aluminum, iron and zirconium is obtained by hydrolysis of their chlorides in a weakly acidic medium, heating the solution to 90-95°C. In the system FeCl₃-AlCl₃-NaCl the

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S/086/60/033/006/001/013
A003/A001

The Chlorine Method in the Industry of Rare and Non-Ferrous Metals

chemical compounds NaFeCl_4 (m.p. 158°C) and NaAlCl_4 (m.p. 152°C) are formed. The compounds can be distilled without decomposition. NaCl forms with the chlorides of niobium and tantalum the following compounds. NaNbCl_6 (m.p. 430°C , decomposition point 287°C) and NaTaCl_6 (m.p. 470°C , decomposition point 370°C). KCl forms with NbCl_5 the compound KNbCl_6 (m.p. 365°C , decomposition point 562°C). Tantalum pentachloride forms with KCl the compound KTaCl_6 (decomposition point 594°C). It is evident that the different thermal stability can be used for separating these substances. A short review on works concerning the chlorination of concentrates and oxides of rare metals published in 1958-1959 is cited. Chlorination is also used in the production of super-pure tin, chromium, manganese, etc. The chlorination of $\text{Mn}(\text{OH})_2$, MnO , Mn_2O_3 and Mn_2O_4 in a liquid medium was studied. γ - MnO_2 and MnCl_2 were obtained as chlorination products. Gold-containing copper-zinc ore was calcinated at 550°C before chlorination to eliminate sulfur and to transform iron to a less active state. The extraction of copper and zinc reached 8-100%, gold 95% and silver 90%. Selenium, tellurium and some selenides and tellurides can be completely dissolved by chlorination at a temperature of 25°C . Petrova studied the chlorination of SnS_2 and concentrates containing SnS_2 (Ref. 44) showing that stannic chloride and sulfur monochloride are formed. The chlorination of lead

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NOV/NOV/1

The Chlorine Method in the Industry of Rare and Non-Ferrous Metals

zinc concentrates in a medium of sulfur monochloride (Ref. 45) was proposed to convert them into a form facilitating their separation. The reactions take place at 120-138°C. A reactor is used, the lower part of which is filled with sulfur monochloride. The sulfide concentrate heated to 120°C is fed to the upper part. A method was developed for extracting tin from concentrates containing 1-10% of tin (Refs. 46, 47). The synthesis of tin chloride directly from tin metal and gaseous chlorine was also developed (Ref. 49). In nickel metallurgy chlorination is used for separating cobalt and nickel (Refs. 50, 51) and for extracting nickel from pure nickel ores. The application of chromium chlorides to obtain chromium metal of high purity was described by Ageyev, Tavadze and Kartvelishvili (Ref. 57). There are 2 tables and 57 references: 45 Soviet, 6 English, 3 American, 1 French, 1 Swiss and 1 Japanese.

SUBMITTED: March 28, 1960

Card 4/4

S/078/61/006/004/013/018
B107/B218

AUTHORS: Morozov, I. S., Simonich, A. T.

TITLE: Thermal and tensimetric investigation of the systems
 $\text{TaCl}_5 - \text{FeCl}_3 - \text{NaCl}$ and $\text{ZrCl}_4 - \text{FeCl}_3 - \text{NaCl}$

PERIODICAL: Zhurnal neorganicheskoy khimii, v. 6, no. 4, 1961, 937-943

TEXT: Three cuts and a number of additional points of the ternary system $\text{TaCl}_5 - \text{FeCl}_3 - \text{NaCl}$ were investigated (Fig. 1). The following points of four-phase equilibria were determined: E_1 with about 2 mole% of TaCl_5 , 48 mole% of NaCl , and 50 mole% of FeCl_3 at 145°C ; E_2 with 2 mole% of TaCl_5 , 47 mole% of NaCl , and 51 mole% of FeCl_3 at 144°C , and the peritectic point P with 3 mole% of TaCl_5 , 49 mole% of NaCl , and 48 mole% of FeCl_3 at 147°C (Fig. 2). The system $\text{TaCl}_5 - \text{NaFeCl}_4$ constitutes a quasi-binary cut. The vapor pressure was determined for three mixtures at temperatures between 150 and 500°C . For a mixture of 24.4 mole% of TaCl_5 , 18.5 mole%

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Thermal and tensimetric investigation...

S/078/61/006/004/013/018

B107/B218

of FeCl_3 , and 57.1 mole% of NaCl , the vapor pressure of TaCl_5 may be expressed by the equation $\log p = -\frac{3180}{T} + 7.5$. A vapor pressure of FeCl_3 is practically absent. It follows from the tensimetric investigations that pure Tantalpentachlorid free from iron or aluminum chloride can be obtained by distillation in the presence of sodium chloride. The boundary system $\text{FeCl}_3 - \text{ZrCl}_4$ and six cuts of the system $\text{ZrCl}_4 - \text{FeCl}_3 - \text{NaCl}$ were studied. At 300°C , this system forms a eutectic with 20 wt% of ZrCl_4 and 80 wt% of FeCl_3 . In the ternary system, the following points of four-phase equilibria were determined (Fig. 3): E_2 with 28 wt% of NaCl , 68 wt% of FeCl_3 , and 4 wt% of ZrCl_4 at 135°C ; E_1 with 8 wt% of ZrCl_4 , 68 wt% of FeCl_3 , and 24 wt% of NaCl at 147°C ; E with 9 wt% of ZrCl_4 , 70.5 wt% of FeCl_3 , and 20.5 wt% of NaCl at 145°C . Tensimetric measurements of three samples ($300-690^\circ\text{C}$) showed that it is not possible to distill zirconium chloride free from ferric chloride by adding NaCl . B. G. Korshunov is

Card 2/5

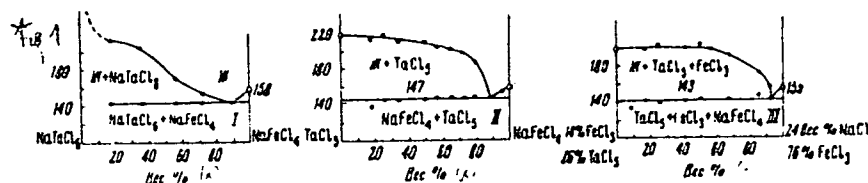
Thermal and tensimetric investigation...

S/078/61/006/004/013/018
B107/B218

mentioned. There are 3 figures, 4 tables, and 8 references: 6 Soviet-bloc. The two references to English-language publications read as follows: C. M. Cook. Ir. J. Amer. Chem. Soc., 81, no. 3, 535 (1959); H. F. Johnston, H. C. Weingartner, W. E. Winsche, J. Amer. Chem. Soc., 64, 241 (1942).

SUBMITTED: July 20, 1960

Fig. 1: Inner cuts of the system $\text{TaCl}_5 - \text{FeCl}_3 - \text{NaCl}$. Legend: x) wt%;
a) liquid.



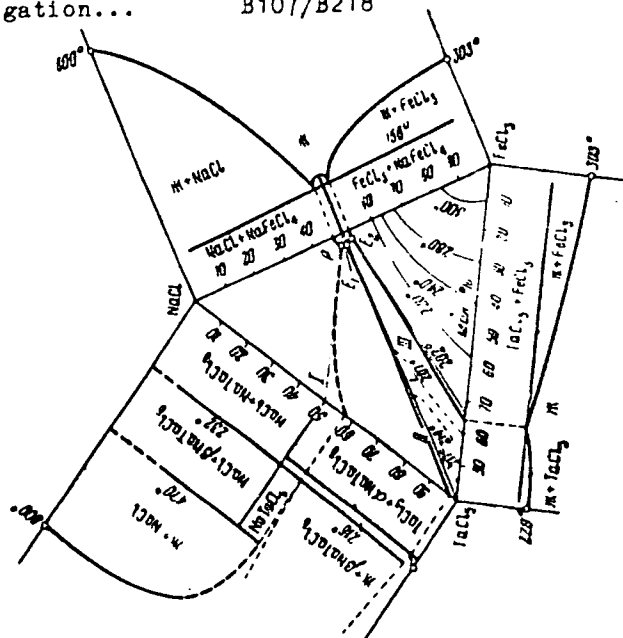
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Thermal and tensimetric investigation...

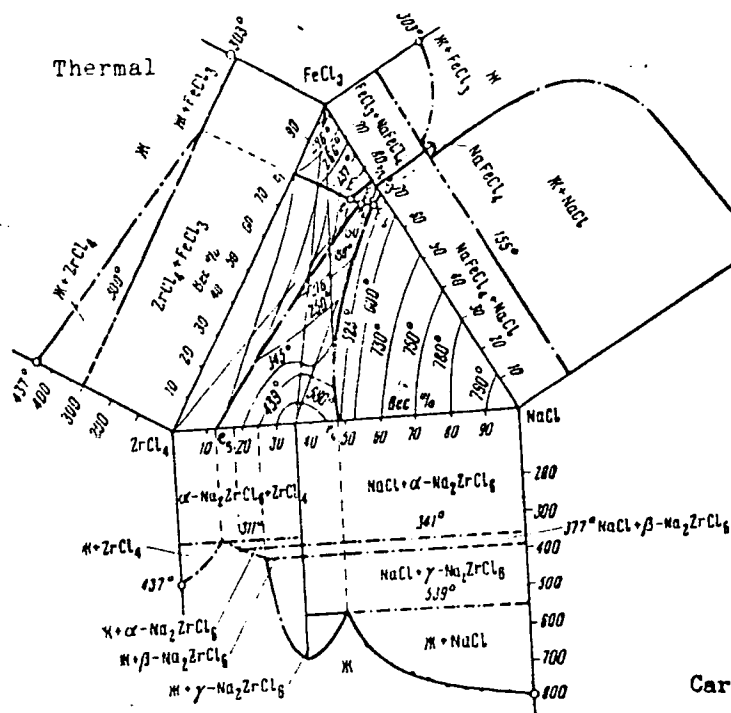
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Fig. 2: Phase diagram of the
syst. $\text{TaCl}_5 - \text{FeCl}_3 - \text{NaCl}$.

Legend: x) mole%; ж) liquid.



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Card 5/5

TOPTYGINA, G.M.; MOROZOV, I.S.

Roentgenographic examination of tetravalent titanium chlorohydroxo
compounds. Zhur.neorg.khim. 6 no.6:1479-1480 Je '61.
(MIRA 14:11)

(Titanium compounds)

TOPTYGINA, G.M.; MOROZOV, I.S.

S/078/61/006/011/007/013
B101/B147

AUTHORS: Morozov, I. S., Toptygina, G. M., Lipatova, N. P.

TITLE: Investigation of compounds formed by titanium trichloride with chlorides of alkali metals and ammonium chloride

PERIODICAL: Zhurnal neorganicheskoy khimii, v. 6, no. 11, 1961, 2528-2535

TEXT: A special problem of producing titanium metal from chloride melts by electrolysis is studied. Conditions under which compounds of $TiCl_3$ with $MeCl$ ($Me = Cs, Rb, K, Na, NH_4$) form, the composition of these compounds, and their solubility in HCl are examined. These compounds which formed in aqueous solution were compared with those forming in salt melts. In the systems $TiCl_3 - MeCl - H_2O$ saturated with $TiCl_3$ and $MeCl$ at $0^\circ C$, no interaction could be determined visually. When saturating the solutions with HCl gas, light-green precipitations deposited which were analytically identified as pentachloro-aquotitanates ($Cs_2TiCl_5H_2O$, $Rb_2TiCl_5H_2O$, $(NH_4)_2TiCl_5H_2O$, $K_2TiCl_5H_2O$). They have a high hygroscopicity

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Investigation of compounds formed by...

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B101/B147

and are attacked by O_2 . The potassium compound immediately decomposes in the air while the sodium compound does not form. Birefringence is characteristic of the Cs-, Rb-, and NH_4 compounds.

Compound	N_1	N_2
$Cs_2TiCl_5H_2O$	1.678 ± 0.002	1.645 ± 0.002
$Rb_2TiCl_5H_2O$	1.682 ± 0.001	1.638 ± 0.003
$(NH_4)_2TiCl_5H_2O$	1.694 ± 0.002	1.664 ± 0.002

Solubility of pentachloro-aquatitanates (% of $TiCl_3$) for ~44 % HCl is 0.35 for the Cs compound, 0.26 for the Rb compound, 0.07 for the ammonium compound, and 1.50 for the K compound. With decreasing HCl concentration, solubility of pentachloro-aquatitanates increases with simultaneous decomposition. In the solid phase, alkali chlorides and (for the Cs compound) $TiCl_3 \cdot 6H_2O$ occur besides the complex compound. In dilute HCl, only the mixtures of alkali- and titanium chlorides precipitate. Thermogravimetric analysis.

Investigation of compounds formed by ..

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B101/B147

graphic and X-ray analyses proved that H_2O of pentachloro-aquatitanates is inside the coordination sphere of the complex. On heating, water is separated. Temperatures: $270^{\circ}C$ for the Cs compound; $212^{\circ}C$ for the Rb compound; $116^{\circ}C$ for the NH_4 compound, and $112^{\circ}C$ for the K compound. In hydrochloric solution, titanium has the coordination number 6. The compounds found in anhydrous systems by other researchers do not form under the conditions described. A paper by M. V. Kamenetskiy (Tsvetnyye metally, 2, 39 (1958)) is mentioned. There are 4 figures, 6 tables, and 14 references: 10 Soviet and 4 non-Soviet. The reference to the English-language publication reads as follows: F. V. Schlossberger, Ind. Eng. Chem., 51 (5), 157 (1959).

SUBMITTED. December 22, 1960

Card 3/3

01078/61/006/011/008/011
P101 B147

AUTHORS Morozov, I. S., Tontyzen, G. M., Lipatova, T. P.

TITLE Thermographic and X-ray analyses of compounds formed from titanium trichloride with chlorides of alkali metals and ammonium chloride

PERIODICAL Zhurnal neorganicheskoy khimii, vol. 6, no. 11, 1961, 2336-2344

TEXT In a previous paper (Zh. neorg. khimii, 6, 2329 (1961)), the authors synthesized the pentachloro aquotitanates of Cs, Rb, NH_4 , and K.

In the present paper, they report on 1) the synthesis of pentachloro titanates of Cs, Rb, NH_4 , and K; 2) the powder patterns of pentachloro aquotitanates and pentachloro titanates, and 3) the thermal analysis of these compounds with Kurnakov's pyrometer. Results: 1) The temperatures at which aquo compounds separate H_2O are: 270°C for $\text{Cs}_2\text{TiCl}_5\text{H}_2\text{O}$,

212°C for $\text{Rb}_2\text{TiCl}_5\text{H}_2\text{O}$, 116°C for $(\text{NH}_4)_2\text{TiCl}_5\text{H}_2\text{O}$, and 112°C for

$\text{K}_2\text{TiCl}_5\text{H}_2\text{O}$. The compounds Cs_2TiCl_5 , Rb_2TiCl_5 , $(\text{NH}_4)_2\text{TiCl}_5$, and K_2TiCl_5

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B101/B147

Thermogravimetric and X-ray analyses of

were obtained by TGA or heating in FCl stream under slow temperature elevation from 100 to 300°C with the Cs compound, from 200 to 400°C with the Rb compound, and from 100 to 150°C with the NH₄ and K compounds.

The light-green color of the initial compounds changed into olive-green with Cs and Rb compounds, and gray with the K compound. The NH₄ compound

remained light-green. 2) The powder patterns of these compounds are shown in Fig. 1. They differ from those of pentachloro aquotitanates, which are also given. Lines of components do not occur in either of the two powder patterns. The X-ray data of K₂TiCl₅ differ from those of

J. Eklund et al. (Z. anorg. Chem. 242, 211 (1959)) obtained by another method of synthesis. X-ray analysis reveals that the crystal structure is changed by removing H₂O from inside the coordination sphere of the

complex whereas the skeleton formed by heavy atoms remains almost unchanged. Ti has the coordination number 6. 3) Due to the low stability of the compounds, the thermogravimetric analysis was conducted in an inert gas stream. It showed that all aquotitanates separated water at the given temperature. At a further temperature elevation, interaction

Card 2/4

Thermographic and X-ray analyses of...

S/078/61/006/011/008/013
B101/B147

between free H_2O and Ti compounds caused side reactions by which deciphering of the thermographs was rendered difficult. (b) Pentachloro titanates have two phases, one of which, $TiCl_3$, is disproportionated into $TiCl_2$ and $TiCl_4$. NH_4 of the NH_4 compound is decomposed, and titanium nitride forms. A paper by M. V. Kamenetskiy (Tsvetnyye metally, 2, 3) (1958)) is mentioned. V. G. Kuznetsov is thanked for advice, and Z. V. Popova for assisting with the X-ray analysis. There are 2 figures, 3 tables, and 25 references: 12 Soviet and 13 non-Soviet. The three most recent references to English-language publications read as follows: K. Komareck, P. Herasymenko. J. Electrochem. Soc., 105, 216 (1958); F. V. Schossberger. Ind. Eng. Chem., 51, 157 (1959); H. P. Klug, E. Kummer, A. Leroy. J. Amer. Chem. Soc., 70, 3064 (1948).

ASSOCIATION: Institut obshchey i neorganicheskoy khimii im. N. S. Kurnakova Akademii nauk SSSR (Institute of General and Inorganic Chemistry imeni N. S. Kurnakov of the Academy of Sciences USSR)

SUBMITTED: February 24, 1961
Card 3/4 3

S/078/61/006/012 108/107
B124/B110

AUTHORS: Morozov, I. A., Tsegledi, L.

TITLE: Thermal and tensimetric studies of the systems ZrCl_4 - KCl ,
 KCl and ZrCl_3 - FeCl_3 - KCl

PERIODICAL: Zhurnal neorganicheskoy khimii, v. 6, no. 12, 1961, p. 2200-2204

TEXT: This paper deals with the chemical interaction of Zr , Al , Fe , and K chlorides on co-crystallization from the melt, which is important to the interpretation of physico-chemical processes which take place during the condensation, separation, and purification of ZrCl_4 from other chlorides. In the course of the study of ternary systems, the binary system FeCl_3 - KCl , three internal cuts of each ternary system, and a number of additional alloys were investigated; experimental results are given in Tables 1 to 3. From the phase diagrams of the two above-mentioned ternary systems, the solidification curves and the boundary lines of the primary crystallization fields for KCl , K_2ZrCl_6 , ZrCl_4 , KAlCl_4 , and AlCl_3 .

Card 1/12

Tigra, and *tematic*

51078/51005 012 007
2126/2110

AS follows: Institut Chimiea Bucuresti, 136, Spl. Independentei, Bucuresti, Romania; Akademiia Nauk SSSR, Institut of General and Inorganic Chemistry, imeni N. S. Kurnakov of the Academy of Sciences of the USSR); Institut chimii Akademii Nauk Rumynskoy Fed. Rep. (Romanian Republic), Kluzhskiy filial (Institute of Chemistry of the Academy of Sciences of the Rumanian People's Republic, 140 Branch).

RECEIVED: May 10, 1961

Table 1. Results of thermal analysis of the system $\text{FeCl}_3\text{-KOH}$.

Legend (A) % by weight; (B) mole; (C) critical thermal point.

Table 1. Results of thermal analysis of the system $\text{IrCl}_3 \cdot \text{AlCl}_3$ in N_2 .

Legend: a - internal cut $\text{KAlCl}_4\text{-ZrCl}_4$; (B) - by weight; (C) - by volume; (D) - molar point; (E) - internal cut $\text{KAlCl}_4\text{-ZrCl}_4$; (F) - molar point.

2000

Thermal and tensile properties

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3124/B110

at $K_2ZrCl_6-AlCl_3$; (G) additional points of ternary alloys.

Fig. 1. Results of thermal analysis of the system $ZrCl_4-FeCl_4-KCl$.

Legend: (A) cut I ($K_2ZrCl_6-KFeCl_4$); (B) % by weight; (C) mole.

(D) critical thermal point, °C; (E) cut II ($ZrCl_4-KFeCl_4$); (F) cut III ($K_2ZrCl_6-FeCl_4$); (G) additional points of ternary alloys in the system $ZrCl_4-FeCl_4-KCl$.

Table 1. Data representing the compound $KFeCl_4$.

Legend: (A) not established

Table 2. Vapor pressure above a chloride mixture of the system $ZrCl_4-AlCl_3-KCl$.

Legend: (A) alloy of 12.23 moles of $ZrCl_4$, 11.34 moles of $AlCl_3$.

Card 4.2

Thermal and tensimetric...

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B124/B110

44.43 mole% of KCl; (B) alloy 2: 17.66 mole% of $ZrCl_4$, 35.30 mole% of $AlCl_3$, 47.0 mole% of KCl.

Table 6. Vapor pressure above a chloride mixture of the system $ZrCl_4$ - $FeCl_3$ -KCl.

Legend: (A) alloy 3: 22.22 mole% of $ZrCl_4$, 33.34 mole% of $FeCl_3$, 44.44 mole% of KCl; (B) alloy 4: 18.04 mole% of $ZrCl_4$, 34.95 mole% of $FeCl_3$, 47.01 mole% of KCl.

Table 4

$t, ^\circ C$	300	310	465	505	555	600	650	700	750	770	800	820	840	850	930
P_{KFeCl_4}	не обнаруж.			0,9	2,1	6,5	10,7	17,3	34,5	44,4	68,2	88,8	110,7	118	210,5
$lg P_{KFeCl_4}$				1,95	0,32	0,81	1,03	1,24	1,54	1,65	1,83	1,95	2,04	2,07	2,3
$\frac{1}{T} \cdot 10^3$				1,3	1,21	1,14	1,08	1,03	0,98	0,96	0,93	0,91	0,90	0,89	0,8

Card 5/22

S/030/61/000/002/002/011
B105/B206

AUTHORS: Yevdokimov, V.I., Candidate of Chemical Sciences
Morozov, I.S., Candidate of Chemical Sciences

TITLE: Application of chlorine in tin metallurgy (Physicochemical
fundamentals of chloridizing at low temperatures)

PERIODICAL: Vestnik Akademii nauk SSSR, no. 2, 1961, 44 - 47

TEXT: Chloridizing of tin at low temperatures, for the economic exploitation of tin ores and concentrates with a tin content below 10%, was investigated by the authors. At present, such ores are not processed at all or only with great losses of tin (up to 50%). A further progress in the tin industry can only be made by applying completely new processes based on new chemical reactions. A sufficiently high yield of tin from concentrates containing less than 10% tin, as well as the separation of tin from all residual components must be warranted. The chloridizing of the powdery charge at low temperatures (120 to 180°C) takes place with the formation of stannic chloride: $\text{Sn} + 2\text{Cl}_2 = \text{SnCl}_4$. The most important investigation

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B105/B206

Application of chlorine ...

results, on the basis of which the chloridizing method at low temperatures was elaborated, are checked next. Under usual conditions, the first stage of the reaction, the formation of SnCl_2 , cannot be observed, since it is very easily oxidized to SnCl_4 by chlorine. Oxidation already takes place at a pressure of the chlorine above the SnCl_2 of 10^{-14} mm Hg. The authors also investigated the kinetics of the process in the kinetic as well as diffusion range. At an increase of the linear chlorine rate from 20 to 70 cm/min, the rate of chloridizing increases from 2 to 6 g tin per hour per cm^2 of the reaction area. An increase of the linear chlorine rate above 70 cm/min does not affect the rate of chloridizing, but the percentage of the utilization of chlorine is reduced (Fig. 1). The chloridizing rate as a function of the temperature is shown in Fig. 2. The equation $\text{SnCl}_4 + \text{Sn} = 2\text{SnCl}_2$ shows the mechanism and kinetics of the interaction of stannic chloride with metallic tin. It was established that this reaction does not start at 600°C as previously assumed, but at 230°C . This fact as well as

Card 2/5

S. 030/61/000/002/002/011
B105/B206

Application of chlorine ...

the determination of the reaction rate at various temperatures and pressures of the SnCl_4 vapor made it possible to elaborate the method of direct SnCl_2 synthesis from chlorine and tin, as well as the method of chlorine-refining of the tin from lead. The losses of tin were considerably reduced and the refining process was intensified. At present, there exist two variants for the production of anhydrous SnCl_2 : at a temperature above 606°C and at one slightly higher than the fusing temperature of tin. On the basis of this method, anhydrous SnCl_2 can be produced in great quantities and without losses. The new method for tin production from concentrates with low tin content consists of the following: the charge of concentrate with low tin content, and coal is heated to $820 - 860^\circ\text{C}$ in a reducing atmosphere. The tin is thereby reduced to metal and remains in the charge in the shape of smallest metal reguli. The reduced charge is cooled and chloridized by means of chlorine gas at 120 to 180°C . Pure metallic tin is produced therefrom. The raw material deposits worth mining increase by more than the double by using this method, and the tin yield increases by 15 to 20%. The processes recommended may easily be automated.

Card 3/5

Application of chlorine ..

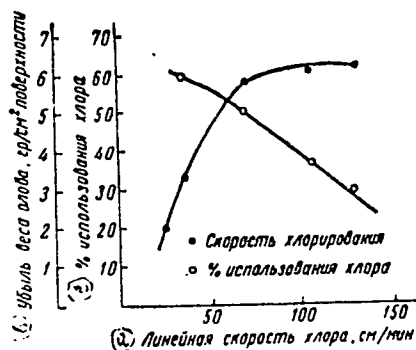
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3105,3206

Since chloridizing proceeds at low temperature, the separation of tin from steel. Collaborators of the Institut obschey i neorganicheskoy khimii Akademii nauk SSSR (Institute of General and Inorganic Chemistry of the Academy of Sciences USSR), TsNTICLOVO ((Tsentral'nyy nauchno-issledovatel'skiy institut olova)(Central Scientific Tin Research Institute)) and Novosibirskiy olovozavod (Novosibirsk Tin Plant) conducted experiments in enlarged installations. It is assumed that the introduction of this method will permit to increase the amount of tin produced and reduce the production costs considerably. There are 2 figures and 3 Soviet references.

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Application of chlorine ...

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3105/3206



Legend to Fig. 1: ● rate of chloridizing, ○ and c) percentage of chlorine utilization; a) linear chlorine rate in cm/min; b) reduction of the tin weight in g/cm² of the area.

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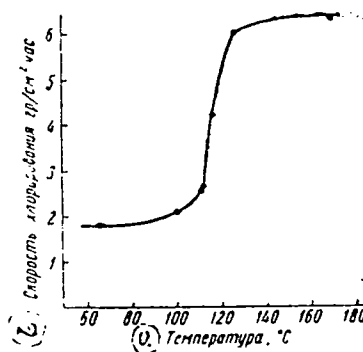


Fig. 2: a) temperature in °C; b) rate of chloridizing in g/cm² hr.

S/080/61/034/004/001/012
A057/A129

AUTHORS: Toptygin, D. Ya., Morozov, I. S.

TITLE: Application of the physico-chemical analysis to solution of the problem of manufacture of titanium and its compounds by the chlorination method.

PERIODICAL: Zhurnal prikladnoy khimii, v. 34, no. 4, 1961, 713 - 725

TEXT: In the present paper a review of Soviet and non-Soviet investigations concerning the reaction of 2-, 3-, and 4- valent titanium with alkali, alkali earths and several other elements is given and the presented results are discussed. The topic of discussions is the interaction of titanium with other elements in chloride systems. The data are of importance for the development of purification methods of titanium tetrachloride, as well as for separation of valuable impurities in titanium processing (such as niobium and tantalum). The impurities present in titanium tetrachloride pass into the latter during reduction. The prevailing method is the purification of $TiCl_4$ with NaCl only as described by A.W. Henderson et al. (Ref. 15: Ind. Engl. Chem., 50, 611, 1958), or

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in mixture with other chlorides as recommended by other authors. The present authors suggested (Ref. 12: Soviet patent no. 133866, February 2, 1960) purification of titanium and niobium chlorides from zirconium, aluminum and iron chlorides by passing salt mixtures of these chlorides through briquettes formed of carbon and alkali chloride. In the present paper the following systems are discussed and corresponding literature data are cited: $\text{TiCl}_4\text{-VOCl}_3$ and $\text{TiCl}_4\text{-VOCl}_4$ were investigated, among others, by the present authors and absolute immiscibility of the chlorides was observed. Corresponding data were obtained by V. Glikman and S. Aftalion-Hinl (Ref. 23: Monatsh., 84, 207, 1956), H. Nishida and K. Oyama (Ref. 25: J. Chem. Soc. Japan, Ind. Chem. Sect., 60 (11), 1434, 1957), J. Snellendon and S. Tyree (Ref. 26: J. Am. Chem. Soc., 81 (10), 2290, 1959), and P. Errlich and W. Siebert (Ref. 27: Z. Anorg. Chem., 301, (5-6), 275 (1959)). The systems $\text{TiCl}_4\text{-SiCl}_4$, $\text{TiCl}_4\text{-CCl}_4$ and $\text{TiCl}_4\text{-C}_6\text{Cl}_6$ were first studied by N. Nasr (Ref. 28: Bl. Chem. Soc. Japan, 8, 195, 1933) and later by H. Sackmann et al. (Ref. 29: Z. Anorg. Chem., 294 (3-4), 120, 1958), and N.N. Delarov et al. (Ref. 22: Izv. AN SSSR, OTN Metall. i topl., 4, 33 (1960)). Systems $\text{TiCl}_4\text{-AlCl}_3$ and $\text{TiCl}_4\text{-FeCl}_3$ were first investigated by L.N. Byngorn (Ref. 32: Ukr. Khim. Zh., 16 (4), 404, 1950), and in more detailed investigations by the present authors (Ref. 34: ZnNKr,

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2(8), 1915, 1957, and Ref. 35: ZhNKh, 2(9), 2129, 1957) by I. Krehma and J. Schau-
mann (Ref. 33: US patent 2502327, 1950), and lately by J. Moriyam and H. Inagaku
(Ref. 38: J. Mining. a Metallurg. Inst. Japan, 76, 101, 1960) P. Ehrlich and G.
Dietz (Ref. 39: Z.Anorg.Ch., 105, 158, 1960), and J. Saheki and K. Funaki (Ref. 40:
J. Chem. Soc. Japan, Pure chem. sect. (Nippon Kagaku Zasshi) 78(6), 754, 1957).
These two systems are of the eutectic type. Solubility of FeCl_3 in TiCl_4 is very
low at room temperature but increase sharply above 204°C . In the systems TiCl_4 -
 NbCl_5 and TiCl_4 - TaCl_5 solubility of NbCl_5 and TaCl_5 in TiCl_4 was determined first
by D. M. Tarasenkova and A. V. Komarova (Ref. 41: ZnOKh, 10(14), 1319, 1940). The
present authors estimated (Ref. 34) that these two systems are of the eutectic
type. This was proved by data from L. A. Nisel'son and G. L. Perekhrest (Ref. 42:
ZhNKh, 3(9), 2150, 1958, and Ref. 39). In presence of NbOCl_3 solubility of NbCl_5
in TiCl_4 changes considerably. Investigations of the present authors (Ref. 34
and 35) into systems TiCl_4 - AlCl_3 - FeCl_3 and TiCl_4 - NbCl_5 - TaCl_5 demonstrated consi-
derable increase in FeCl_3 solubility in TiCl_4 in presence of AlCl_3 . Obtained data
also indicated that recommendation for separation of NbCl_5 and TaCl_5 based on
different solubility in TiCl_4 (Ref. 41) and analogous separation of Al and Fe
(Ref. 33) are unfounded, since from TiCl_4 solutions solid chloride solutions

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with the initial composition with a separate tit. In systems $TiCl_4-NbCl_5-AlCl_3$, $TiCl_4-NbCl_5-FeCl_3$ and $TiCl_4-AlCl_3-TaCl_5$ S. Marozov observed (Ref. 41: ZHUK, 1(12) 2792, 1956) a sharp increase in solubility of $NbCl_5$ and $TaCl_5$ in $TiCl_4$ in presence of $AlCl_3$ and $FeCl_3$ but also a sharp increase in solubility of the latter effected by the presence of $NbCl_5$ and $TaCl_5$. Solubility of $TiOCl_2$ in $TiCl_4$ was investigated by T. A. Zavaritskaya and T. A. Pustovalova (Ref. 48: Tsvet. met., 10, 50, 1958). (Ref. 22) and D. Ya. Toptygin (Ref. 47: Dissertation, M., 1960). According to this data it can be assumed that $TiCl_2$ impurities in $TiCl_4$ distillates are caused by moisture penetrated into the condensation device. Since commercial $TiCl_4$ contains gaseous impurities, data on solubility of gases in $TiCl_4$ presented by Delanova (Ref. 22) are important. Literature data demonstrated that chlorides which are not present in technical $TiCl_4$, such as $HgCl_2$, Hg_2Cl_2 , $GaCl_3$, $MoCl_5$, WCl_6 , $AsCl_3$, $SbCl_3$, $SbCl_5$, $SeCl_4$, $TeCl_4$, $MnCl_2$ etc., are soluble in $TiCl_4$, but do not form compounds, while sulfur and phosphorus chloride form compounds with $TiCl_4$. $SnCl_2$ and $ZnCl_2$ as observed by Toptygin (Ref. 47), are very different from the other chlorides in relation to $TiCl_4$. Interaction between $TiCl_4$ and alkali and alkali earth chlorides have been investigated frequently, because of the importance for the purification procedures of

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$TiCl_4$. Corresponding experiments were made by Ehrlich (Ref. 59: Z. Naturforsch., 9B (4), 326, 1954), S.F. Belov and S. I. Sklyarenko (Ref. 60: Tsvet. met., 11, 37, 1958), and L. A. Tsiolkina, M. V. Smirnov (Ref. 61: ZhNKh, 4(1), 158, 1959). F. V. Schosstenger proposes (Ref. 54: Chem.Eng. Prog., 53, 94, 1957); Ref. 55: US Patent 2857242, October 21, 1958; Ref. 56: US patent 2857143, October 21, 1958; Ref. 57: US patent 2857265, October 21, 1958; Ref. 58: Ind. Engl. Chem., 51(5), 669, 1959) a large scale production of pure $TiCl_4$ by decomposition of potassium or ammonium chloro-titanates. The present authors prepared (Ref. 62: ZhNKh, 5(1), 88, 1960) alkali hexachloro-titanates directly by reaction of gaseous $TiCl_4$ with alkali metal chlorides. S. N. Flengas (Ref. 63: Ann. N. Y. Acad. Sci., 79, (1), 853, 1960) synthesized K_2TiCl_6 by the reaction of $TiCl_4$ with KCl, as well as $TiCl_4$ with KCl + NaCl, and agrees contrary to the opinion of the present authors that above 800°C Na_2TiCl_6 is formed. Density of the saturated vapor of $TiCl_4$ over K_2TiCl_6 was determined among others by (Ref. 59) and S.N. Flengas (Ref. 63: Canad. J. Chem., 38(6), 812, 1960). Thermal stability of alkali and ammonium hexachlorotitanates was studied by the present authors (Ref. 70: ZhNKh, 5(1), 2518, 1960) and it was observed that none of these titanates is stable at 700-800°C. In this work (Ref. 70) the authors were first to describe a new type of

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titanium compounds obtained by partial hydrolysis of alkali hexachlorotitanates. The physico-chemical principles of $TiCl_4$ purification from $AlCl_3$ and $FeCl_3$ by means of alkali metal chlorides is based on the fact that $TiCl_4$ does not form compounds with $NaCl$; it forms unstable compounds with KCl , which decompose at relatively low temperatures. H. Johnstone et al. (Ref. 79: J. Am. Chem. Soc., 64, 241, 1942) determined that the system $FeCl_3$ - $NaCl$ has one eutectic point. Contrary to the opinion of the last-mentioned author the present authors asserted (Ref. 47: Ref. 80: Izv. AN SSSR, Otkryt. Nauch. 1959) the formation of $NaFeCl_4$, which does not decompose in $TiCl_4$ - $FeCl_3$ - $NaCl$ contains sufficient $NaCl$ for the formation of $NaFeCl_4$, the $TiCl_4$ phase does not contain even traces of $FeCl_3$, while in excess of $FeCl_3$ a part of the latter is dissolved in $TiCl_4$. The use of $NaCl$ for $TiCl_4$ purification can be applied also to $AlCl_3$, since $NaAlCl_4$ (as well as $NaFeCl_4$) is formed at relatively low temperatures and has a low vapor pressure up to $500^\circ C$. According to V.G. Goplyenko and A.I. Ivanov (Ref. 55: Izv. AN SSSR, OTN, Metallurg. i topl., 4, 1950) which studied the system $TiCl_4$ - $AlCl_3$ - $NaCl$, the $NaAlCl_4$ phase can dissolve 2% of $TiCl_4$. Several investigations demonstrated that $NbCl_5$ and $TaCl_5$ form in presence of $TiCl_4$ the compounds $NaNbCl_6$ and $NaTaCl_6$. The latter decompose at low temperatures, but the reaction occurs with $NaCl$ at

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higher temperatures than between FeCl_3 or AlCl_3 and NaCl . In TiCl_4 purification processes NaCl can be substituted by KCl or NH_4Cl . Contrary to Me_2TiCl_6 compounds chlorides of 2- and 3-valent titanium are more stable. Systems $\text{TiCl}_3\text{-NaCl}$ and $\text{TiCl}_3\text{-KCl}$ were studied by M.V. Kamenetskiy (Ref. 88: Tsevt. met., 2, 39, 1958); Ref. 89: Izv. vuzov, Tsvet. metallurgiya, 3(1), 119, 1960) and the congruent melting compound K_3TiCl_6 was observed, while B.F. Markov and R. V. Chernov (Ref. 90: Ukr. Khim. zh., 25(3), 279, 1959) estimated also KTiCl_4 , and P. Ehrlich et. al. (Ref. 91: Z. Anorg. Ch., 299, (3-4), 213, 1959) determined the incongruent melting compounds Na_3TiCl_6 and K_2TiCl_5 . According to Ref. 90 in the system $\text{TiCl}_3\text{-RbCl}$ and $\text{TiCl}_3\text{-CsCl}$ congruent melting Rb_3TiCl_6 , RbTiCl_4 , Cs_3TiCl_6 and CsTiCl_4 are formed. K. Komarek and P. Gerasimenko (Ref. 93: J. Electrochem. Soc., 105 (4), 210, 1958) studied the systems $\text{TiCl}_2\text{-NaCl}$, $\text{TiCl}_2\text{-KCl}$ and $\text{TiCl}_2\text{-MgCl}_2$ and determined the two chemical compounds NaTiCl_3 and Na_2TiCl_4 while P. Ehrlich and H. Kühnel (Ref. 94: Z. anorg. Ch., 292, 146, 1957) observed KTiCl_3 and K_2TiCl_4 . The system $\text{TiCl}_2\text{-MgCl}_2$ was investigated by K. Komarek and P. Gerasimov (Ref. 95: J. Electrochem. Soc., 105, (4), 210, 1958) and no chemical compounds were observed. M. Siebert and M.A. Steinberg (Ref. 96: J. Metals., 8(9), 1162, 1956) indicate that TiCl_2 is soluble in molten lithium chloride. There are 2 tables and 96 references: 49 Soviet-bi and 57 non-Soviet-bloc.

SUBMITTED: November 21, 1960
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AUTHORS: Morozov, I. S., Krozhin, V. A.

TITLE: Thermal and tensimetric investigations of the systems NbCl_5 - AlCl_3 - KCl , KNbCl_6 - KTaCl_6 - KAlCl_4 , and NbCl_5 - KTaCl_6 - KAlCl_4 .

PERIODICAL: Zhurnal neorganicheskoy khimii, v. 7, no. 10, 1961, pp. 1711-1714

TEXT: The following binary systems were newly constructed: KAlCl_4 - KAlCl_4 with a single eutectic at 180°C (composition in mole %: KAlCl_4 4.7); KTaCl_6 - KNbCl_6 forming a complete series of solid solutions; KAlCl_4 - KNbCl_6 with a single eutectic at 201°C (composition in mole %: KAlCl_4 55, KNbCl_6 45); KTaCl_6 - NbCl_5 with a single eutectic at 180°C (composition in mole %: KTaCl_6 55, NbCl_5 45); KAlCl_4 - NbCl_5 with a single eutectic at 170°C (73 mole % NbCl_5) and demixing as from 79 mole % NbCl_5 . The ternary system NbCl_5 - AlCl_3 - KCl has the following eutectic triple points:
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4-phase equilibrium	Composition, mole%			Melting point, °C
	KCl	NbCl ₅	AlCl ₃	
E ₁ liq ⇌ AlCl ₃ + NbCl ₆ + KAlCl ₄	5	45	50	115
E ₂ liq ⇌ KCl + KNbCl ₆ + KAlCl ₄	52	33	15	175
E ₃ liq ⇌ KNbCl ₆ + KAlCl ₄ + NbCl ₅	6	90	4	160

In the quasi-ternary system KNbCl₆-KTaCl₆-KAlCl₄, a eutectic was found in the 3-phase equilibrium liq ⇌ KAlCl₄ + solid solution KTaCl₆ + KNbCl₆; this eutectic corresponds to the mole% composition KAlCl₄ 36, KNbCl₆ 41, KTaCl₆ 33, and to a melting point of 170°C. Polymorphic decomposition of the hexachlorides was not observed. The liquidus surface shows two crystallization ranges (KAlCl₄ and the solid solutions KNbCl₆, KTaCl₆).

In the quasi-ternary system NbCl₅-KTaCl₆-KAlCl₄, a eutectic triple point was determined in the 4-phase equilibrium liq ⇌ KAlCl₄ + KTaCl₆ + NbCl₅; this

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point corresponded to the mole% composition $KAlCl_4$ 16.5, $NbCl_5$ 5.2, $KTaCl_6$ 26. and to a melting point of $170^\circ C$. The liquidus surface shows ranges corresponding to the crystallization of the three components with a demixing in the $NbCl_5$ range. The following systems were studied tensimetrically: (1) $NbCl_5$ - $AlCl_3$ - KCl ; (2) $NbCl_5$ - $TaCl_5$ - $AlCl_3$ - KCl ; (3) $NbCl_5$ - $TaCl_5$ - $AlCl_3$ - $NaCl$. (1) The vapor pressure of $NbCl_5$ drops considerably with rising KCl content of the melt. (2) and (3) The concentration of $TaCl_5$ increases more than that of $NbCl_5$. There are 13 figures and 11 tables.

SUBMITTED: November 15, 1961

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